Evaluation of the performance of encapsulated lifting system composting technology with a GORE(R) cover membrane: Physico-chemical properties and spectroscopic analysis

Mutaz Al-Alawi^{1†}, Loubna El Fels^{2,3}, Ramadan Benjreid¹, Tamas Szegi¹, Mohamed Hafidi^{2,4}, Barbara Simon¹, Miklos Gulyas¹

ABSTRACT

Composting is among the most effective integrated waste management strategies used to recycle sewage sludge (SS) waste and generate a useful product. An encapsulated lifting system is a relatively new industrial-scale composting technology. The objective of this study was to evaluate the effectiveness of composting dewatered stabilized SS mixed with green waste using this new technology. The composting process was monitored by changes in the physico-chemical properties, UV-visible spectra, and fourier transform infrared (FTIR) spectra. The composting temperature was steady in the thermophilic range for 24 and 12 d in the intensive and maturation phases, respectively, which fulfilled the disinfection requirement. Moreover, the temperature increased rapidly to 76.8°C within three days, and the thermophilic temperatures peaked twice and lasted longer than in traditional composting, which accelerated SS degradation and decreased the composting period necessary to obtain mature compost. FTIR spectroscopic analysis showed a diminished in methyl group derived from methylene C-H aliphatic groups because of organic matter degradation by microorganisms and an increased number of aromatic chains. The new technology may be a viable and sustainable alternative for SS management that converts waste into compost that is useful as a soil amendment.

Keywords: Encapsulated lifting system (ELS) composting, GORE(R) cover membrane, Physico-chemical properties, Sewage sludge, Spectroscopic analysis, Two-stage composting

1. Introduction

Rapid increases in population density and mounting urbanization and industrialization have led to many additional wastewater treatment plants being built; as a result, the amount of wastewater and sewage sludge (SS) generated has dramatically increased, and the disposal of these materials has become a major technical challenge in terms of both operating costs and ecological damage. Untreated SS and direct agricultural use without previous treatment and stabilization can potentially cause groundwater pollution, generate odors, and pose risks to human health and the environment because SS contains high concentrations of toxic metals, infectious microbes and organic micropollutants [1]. Moreover, the current capital and operating costs for sludge management can reach 25-50% of the total cost of the wastewater treatment process [2].

The production of SS as a result of urban wastewater treatment in the 27 continental EU countries will reach 13.5 million tons of dry matter by 2020 [3], and the environmental pollution resulting from this SS will become a key issue if it is left untreated. It is therefore important to identify alternative disposal methods and effective technologies for waste reuse to eliminate the harmful



This is an Open Access article distributed under the terms of the Creative Commons Attribution Non-Commercial License (http://creativecommons.org/licenses/by-nc/3.0/) which per-

mits unrestricted non-commercial use, distribution, and reproduction in any medium, provided the original work is properly cited.

Copyright © 2020 Korean Society of Environmental Engineers

Received February 14, 2019 Accepted April 29, 2019

†Corresponding author

Email: alawi1979@yahoo.com

Tel: +36-70-241-2661 Fax: +36-28-410804

ORCID: 0000-0002-8081-1625

¹Department of Soil Science and Agricultural Chemistry, Institute of Environmental Science, Faculty of Agriculture and Environmental Sciences, Szent Istvan University, Hungary

²Laboratory of Ecology and Environment (L2E) (Unit Associated with the CNRST, URAC32), Faculty of Science Semlalia, Cadi Ayyad University, Morocco

³Higher Institute of Nursing Professions and Health Techniques, Marrakech-Safi, Morocco

⁴Agrobioscience Program, Mohammed VI Polytechnic University, Benguerir, Morocco

environmental impacts of this SS.

Composting can help efficiently manage SS by reducing the volume and weight of initial waste by approximately 50%, as well as killing pathogenic microorganisms due to the generation of heat (50°C to 70°C) during its thermophilic phase, and producing mature and stable organic substances [4]. However, to obtain a high-quality compost product, efficient composting technologies must be used [5]. Currently, various composting technologies are available; the choice of technology depends on the operational cost, the time needed to reach compost maturity, the land availability, and the source of raw materials. In recent years, due to the demand for new SS treatment technologies, composting technologies have been subjected to considerable modifications (e.g., cocomposting, vermicomposting, and windrow composting) to make composting technology more cost effective [6].

Two-stage composting is a relatively new concept in the production of compost, which integrates two completely different technologies into a single composting process in order to improve the final compost quality and increase the cost-effectiveness of the process; it also helps to minimize the negative environmental impact of traditional composting technologies. Several two-stage systems have been examined: Some systems combine two composting technologies, such as the integration of in-vessel composting with periodically turned windrow composting or the integration of vermicomposting and traditional composting methods, while other systems include some form of mechanical treatment, such as waste pre-treatment, mechanical biological treatment, or anaerobic digestion, prior to the composting process [7].

The first paper addressing the two-stage composting was by Kulikowska and Klimiuk [8], who studied SS waste. The technology involves a transfer between two systems, typically a periodically turned windrow and an aerated bioreactor. The organic waste spends a short time (10 d) in the aerated bioreactor to allow decomposition and sanitation of the organic material before spending 207 d in the windrow to reach maturity. Another two-stage composting technology involves primary composting (PC) and secondary composting (SC) [9]. During PC, the thermophilic phase (50°C to 60°C) starts quickly and persists for a long period. When the temperature decreases to ambient temperature, PC is considered complete, and SC begins, during which decomposition continues, resulting in a second thermophilic phase [9]. This novel technology thus results in two thermophilic temperature peaks during the whole composting process and a longer thermophilic phase than that obtained with traditional composting (one stage with composting windrows); in addition, compared with traditional composting, the composting duration of the new method is shortened, and the quality and stability of the compost product are increased. Only 30 d are required for the production of mature compost with two-stage composting, as opposed to the 90 d to 270 d required in traditional composting [10].

González et al. [11] demonstrated the advantages of SS composting at the industrial scale by utilizing an aerated static windrow covered with a novel semipermeable film. This novel technology reduces carbon dioxide, sulphur, and nitrogen oxide emissions and also reduces the overall process time by reducing heat losses and prolonging the thermophilic phase; furthermore, by increasing the treatment capacity, this technology increases system efficiency and reduces operating costs [11]. However, the disadvantages of

such systems include complex infrastructure and greater costs than required for open-windrow composting technologies.

The use of a semipermeable cover system with air-floor aeration for a mixture of SS and pruning debris green waste (GW) led to a change in the bacterial profile; microorganisms with the ability to degrade recalcitrant compounds were detected in the early stages of composting, presumably due to the high temperatures rapidly reached during the first few days of composting [12]. In a previous study, during the composting of pig manure and wheat straw using a semipermeable membrane cover with a bottom-up aeration system, the relative abundances of anaerobic Clostridiales and pathogenic Pseudomonas decreased, and the abundance of the facultative anaerobe Cellvibrionales, which has a complex cellulase system, increased [13]. The use of a semipermeable membrane during pig manure and wheat straw composting with a bottom-up aeration system allowed even and sufficient oxygen concentration to be achieved with a slight positive pressure, decreasing the anaerobic zone in the compost pile and decreasing methane (CH₄) emissions by 22.42% [14]. Greenhouse gases (CO₂, CH₄, and N₂O), as well as ammonia production and emissions were also decreased during cattle manure composting using a semipermeable membrane cover [15].

The encapsulated lifting system (ELS) is a relatively new composting technology that includes a two-phase aerated static windrow combined with a GORE(^R) cover membrane and a bottom-up aeration system. However, to date, data on the use of ELS technology with a GORE(^R) cover membrane in SS composting remain unavailable. Therefore, this study is novel, and the goal was to evaluate the effectiveness of this technology based on monitoring the changes in the physico-chemical properties and the UV-visible and fourier transform infrared (FTIR) spectra during a SS composting process.

2. Materials and Methods

2.1. Composting Materials

The study was carried out at a composting facility at the municipal wastewater treatment plant in the city of Kecskemet, Hungary. The plant serves a population of 240,000, treats 20,000 $\rm m^3/d$ and generates 22,400 t/y of activated SS (21-23% dm content). The composting facility was established in 2010, with its initial production capacity being approximately 5,000 $\rm m^3/y$.

The dewatered stabilized SS (DSSS) and GW were used for composting. The DSSS was obtained by centrifuging stabilized sludge from a biogas digester, while the GW (grass, wood chips, tree leaves, etc.) was collected from the parks and gardens in Kecskemet. The DSSS was mixed with the GW materials, which acted as bulking agents, at a 1:4 proportion of sludge to bulking agent to obtain optimum moisture and a C/N ratio of approximately 65% and 25:1, respectively. The main characteristics of the raw materials used are presented in Table 1. The DSSS was characterized by having the highest moisture and nitrogen content, while the GW had a high organic matter (OM) content and the lowest moisture content. The GW materials were thus added to lower the overall moisture content, increase the C/N ratio, and enhance the final structure of the composted material.

Table 1. Characteristics of the Raw Materials Used for Composting

Organic waste	Moisture (%)	Organic matter (%)	Total carbon (%)	Total nitrogen (%)	C/N	EC (mS cm ⁻¹)	pН
DSSS	80.5 ± 0.7	71.8 ± 1.5	31.5 ± 1.2	$4.1 ~\pm~ 0.0$	$7.71 \pm .4$	$3.1 \pm .01$	$7.4~\pm~0.0$
GW	29.4 ± 1.1	97.4 ± 0.2	54.0 ± 1.0	$0.7 ~\pm~ 0.0$	$74.7 ~\pm~ 2.4$	0.7 ± 0.0	$7.1~\pm~0.2$

± represents the standard deviation based on three replicates

2.2. Composting Process

Composting processes were carried out using ELS with a $\mathrm{GORE}(^R)$ cover membrane at an industrial scale (Fig. 1). The composting process consisted of two phases of aerated static windrow treatment under $\mathrm{GORE}^{[R]}$ cover membrane an intensive phase (4 w) and a maturation phase (3 w).

At the beginning of the intensive phase, the mixture was loaded into a composting cell; this was on day 0. The cell (35 m long, 8 m wide, and 2.6 m high, with a 730 m³ total capacity) had three reinforced concrete side walls, was covered with GORE(R) cover membrane, and had a dedicated ventilation system to ensure its oxygen supply. When the windrow temperature dropped to ambient temperature at day 28, the intensive phase was considered complete. At that time, the mixture was removed from the composting cell with an excavator and placed in another composting cell for the maturation phase. That cell (30 m long, 8 m wide, and 2.6 m high, with a 630 m³ total capacity) had three reinforced concrete side walls, which were covered with a GORE(R) cover membrane and had a ventilation system to provide adequate aeration. The maturation phase began on days 29 till 49. A 4 m high glass-fibre tarpaulin was attached to the reinforced concrete walls, and the whole structure was covered with a GORE^(R) cover membrane during both phases to avoid excessive water loss caused by wind and evaporation. The two-stage composting process was considered complete when the temperature of the windrow declined to ambient temperature. The material was then screened using a screening drum with 12 x 12 mm holes.

The temperature was measured using Pt-100 type sensor housed in stainless-steel sheath that were connected to a data acquisition system linked to a standard PC. The Pt-100 sensor was placed in the windrow at measuring points located at half the windrow's height. The daily mean temperatures were calculated based on recorded hourly temperatures that were taken continuously throughout the process using a digital recorder. Another temperature sensor was placed outside to measure the ambient temperature for comparison.

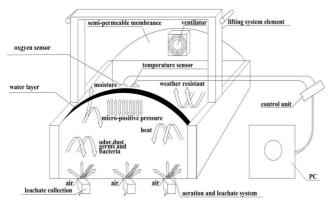


Fig. 1. Schematic diagram of the composting technology.

2.3. GORE(R) Cover Membrane

The GORE(R) cover membrane had three different layers: An expanded polytetrafluoroethylene (ePTFE) membrane/microporous film and two protective layers made of high-robust polyester fabric that formed the laminate's top and bottom layers. The membrane in the middle layer acted as the functional component, and the membrane parameters were as follows: water vapor permeability resistance of $\leq 19.5 \text{ m}^2 \text{ Pa W}^1$, air permeability of 1.5-6.5 m³ m² h⁻¹, and hydrostatic pressure of $\geq 50 \text{ kPa [15]}$.

The GORE($^{\mathbb{R}}$) cover membrane is waterproof, windproof, and reusable; the material is highly tolerant to biological and chemical degradation and to heat (up to 200-260°C) [15]. With a micropore size of approximately $\leq 0.2~\mu m$, the membrane effectively prohibits the release of microbes, gaseous substances, and particulate matter while remaining semipermeable to moisture [16]. Most of the water vapor generated during the early composting phase in response to decomposition of OM did not escape the membrane due to the limited permeability of the membrane, however, building up an aqueous condensate layer on the inner surface of the cover. This aqueous condensate layer acted as a scrubber for odours by dissolving odour compounds and forming droplets that returned the compounds to the composting material where they could be further decomposed by microorganisms.

2.4. Ventilation System

Air was supplied via ventilators and trenches placed at the base of the windrow. These trenches acted as ducts to provide air to the windrow and to collect leachate from the windrow. Aeration was accomplished by ventilators with a maximum flow rate of $2,400~\text{m}^3/\text{h}$ at 2,940~rpm and 80~dB(A), using a forced aeration system placed at the base of the windrow. The ventilator engine power was 2.5~kW. A ventilation system controlled by a timer was used to control the windrow temperature. The injected flow rate diffused the air intermittently. In the intensive phase, the aeration frequency was 491~min/d (491~min ON/949 min OFF), and the air flow rate was $172~\text{m}^3~\text{air/m}^3~\text{compost/d}$, while in the maturation phase, the frequency was 404~min/d (404~min ON/1,036 min OFF), and the air flow rate was $166~\text{m}^3~\text{air/m}^3~\text{compost/d}$.

2.5. Sampling Strategy and Collection

Composting was conducted for 49 d. Homogenous 1-kg samples that represented the average conditions of the entire windrow were collected in accordance with the US Composting Council standard [17] at the following intervals: 0 (first day of composting/initial non-decomposed material), 7, 14, 21, and 28 d during the intensive phase, and 35, 42, and 49 d during the maturation phase.

2.6. Physicochemical Analysis of Compost Samples

Ash content was determined by burning samples (previously dried at 105°C) at 550°C for 7 h in a muffle furnace [17]. OM were estimated as follows [17]:

$$OM (\%) = 100 - ash (\%)$$
 (1)

A 1:10 aqueous extract of fresh compost was used to analyse the pH and electrical conductivity (EC), as described by Guo et al. [18]. Losses of OM due to OM mineralization were calculated based on the initial and final ash content for each time interval according to the equation [19]:

$$OM_{loss}$$
 (%) = 100 - 100 $[X_1(100 - X_2)] / [X_2(100 - X_1)](2)$

where X_1 and X_2 are the initial and final ash concentrations, respectively.

Total nitrogen (TN) and total carbon (TC) were determined by subjecting air-dried samples to dry combustion at a combustion temperature of 1,100-1,200°C using a CNS analyzer (Fisons NA 1500 Series II CNS analyzer). The cation exchange capacity (CEC) of the air-dried samples was determined according to Harada and Inoko [20]. The ammoniacal nitrogen (NH₄ $^+$ -N) and nitrate nitrogen (NO₃ $^-$ -N) were extracted by mixing air-dried samples with 0.5 M K₂SO₄ at a ratio of 1:10 (w/v), mechanically shaking for 1 h, and filtering through 0.45- μ m membrane filters. The filtrate was used for NH₄ $^+$ -N and NO₃ $^-$ -N determination using the colorimetric method, as described by Okalebo et al. [21].

2.7. UV-visible Spectroscopic Analysis

UV-visible spectroscopy analysis was performed according to the following method by Zbytniewski and Buszewski [22]. One gram of air-dried compost and 50 mL of 0.5 M NaOH were shaken for 2 h and then centrifuged at 3,000 rpm for 25 min. The absorbance of the supernatant was measured at $\lambda=280$ nm (A280), 472 nm (A472), and 664 nm (A664) using a UV-2000 spectrophotometer covering a frequency range of 200 nm to 800 nm. The following absorbance ratios were calculated: $E_{2/6}=A280/A664$, $E_{4/6}=A472/A664$, and $E_{2/4}=A280/A472$.

2.8. FTIR Spectroscopic Analysis

Air-dried samples from each composting step were analyzed with a Bruker DRIFT (diffuse reflectance infrared fourier transform) FTIR spectrometer covering a frequency range of 4,000-400 cm⁻¹. The resolution was set to 4 cm⁻¹, and 32 scans were recorded before being averaged for each spectrum and corrected against ambient air, which acted as the background.

2.9. Statistical Analyses

The data shown in the tables and figures represent the means of three replicate subsamples for each composite sample with standard deviations calculated using Microsoft Excel 2016. Each set of three subsamples was prepared from the corresponding composite sample (representing the average conditions of the entire windrow in question) and analyzed independently.

3. Results and Discussion

3.1. Temperature Evolution

The composting process is principally a biological phenomenon that relies highly on temperature fluctuations within windrows. In this case, the mixture of DSSS and GW at the beginning of the composting process was characterized by its large fermentable fraction. On day 0 (the start of the intensive phase), the temperature began to rise rapidly from 31.2°C, reaching a peak temperature of 76.8°C within three days (Fig. 2). These temperatures were higher and increased faster than those observed during composting of the same substrate using classical methods [23, 24]. The micropositive pressure environment resulting from the joint effects of the forced bottom-up ventilation system and the GORE(R) cover membrane was conducive to providing the even supply of sufficient oxygen to microorganisms that is required to degrade OM. Ultimately, the microbial degradation of easily decomposable OM was enhanced, the decomposition rate increased, and a large amount of heat produced. After peaking during the intensive phase, the temperature decreased, remaining in the mesophilic range (below 42°C) between days 25 and 31. The decrease in temperature can be explained by two main phenomena: the exhaustion of the initial simple molecules and the development of conditions hostile to microorganism activity.

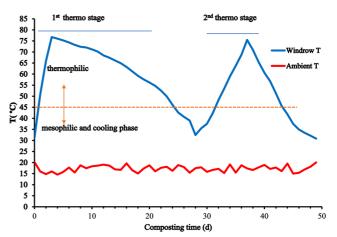


Fig. 2. Evolution of temperature during SS-GW composting.

During the composting process, a paradoxical situation was observed. As part of the maturation phase (beginning on day 29), the temperature began to increase again to produce a second thermophilic phase, with a peak temperature of approximately 75.4°C again achieved on day 37 (Fig. 2). Insam et al. [25] demonstrated that temperatures from 26°C to 45°C lead to favourable conditions for the growth of actinomycetes and other heat-tolerant bacteria, an observation that explains why, during the short mesophilic stage (days 25 to 31), the conditions favoured the regrowth of microorganisms, subsequently leading to the reappearance of microorganism activity and a second increase in temperature. However, after the second thermophilic stage, the temperature progressively decreased to ambient temperature, and the covered compost did not exhibit any large variations in temperature during storage, indicating that the microbial activity and mineralization had ceased

and that humification resumed, demonstrating that the compost was stable and that the process could be considered complete. This phenomenon of two thermophilic peaks has been observed by several authors [26-28]. Zhang et al. [9] subjected raw materials to a two-phase composting process comprised of primary fermentation and secondary fermentation during which a thermophilic phase was attained twice. Tønner-Klank et al. [29] showed that appropriate composting can eliminate many human pathogens in different types of compost, as the temperatures attained (50°C to 70°C) during treatment kill enteric microorganisms.

With regard to the length of thermophilic periods, the composting temperature must exceed 45°C for at least three consecutive days to destroy pathogenic organisms and thus meet disinfection requirements [30]. The results showed that the total duration of thermophilic temperatures in this experiment (intensive phase plus maturation phase) was > 3 d. This finding indicated that the duration of the thermophilic stage was sufficient to kill pathogens and meet the disinfection requirements.

By using the new technology, the temperature remained high and steady in the thermophilic temperature range for 24 d and 12 d in the intensive phase and maturation phase, respectively, as the GORE(R) cover membrane completely enclosed the windrow, retaining heat. This technology resulted not only in more rapid attainment and prolongation of thermophilic temperatures but also in the creation of two thermophilic temperature peaks (one during the intensive phase and one during the maturation phase); the main consequence of this was more rapid decomposition of organic waste. El Fels et al. [24] and El Havany et al. [31] stated that traditional composting requires 180 d and 105 d, respectively, to produce a mature product; two-stage composting consisting of the use of an aerated windrow under a semipermeable film and in the second stage a static open windrow requires 190 d [11]. However, the proposed new technology shortens the SS composting time significantly, producing mature compost in only 49 d.

3.2. Evolution of pH and Conductivity

3.2.1. pH variation

Changes in pH and EC were monitored during the composting of SS mixed with GW. During the first week of composting, the pH value decreased from 7.0 to 6.1 (Fig. 3), which could be due to production of organic acids such as acetic acid and butyric acid from microorganism activity during the degradation of easily degradable compounds, or due to the loss a large quantities of carbon dioxide during the composting process [32]. The decrease in pH could also be due to a reduction in the buffering capacity of the system deriving from volatilization of ammonia in the thermophilic phase [33]. Normally during SS composting processes, NH₃ volatilization takes place coincidentally with high temperatures and lower initial C/N ratios, thereby reducing the agronomic value of the compost and generating undesirable odours [34]. NH₃ loss during composting ranges from 16% to 76% of TN loss [35]; advanced compost technology to reduce TN loss is thus needed. The GORE(R) cover membrane inhibited NH3 emissions as some NH3 absorbed by the water layer created on the inner surface of the cover, and as a result nitrogen conservation was achieved through the transformation of reduced ammonia [15].

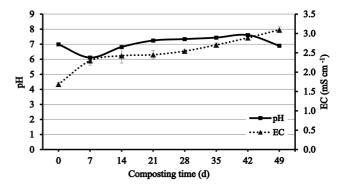


Fig. 3. Evolution of pH and EC during composting.

The increase in pH from 6.1 to 7.3 (Fig. 3) during the ensuing 3 w of composting (d 7 to 28 of the intensive phase) can be explained by the production of ammonia during ammonification and the mineralization of organic nitrogen, which releases those bases already present in organic waste [24]. Vergnoux et al. [36] showed that a higher oxygen concentration resulted in more rapid acid decomposition and thus a faster increase in pH. Baeta-Hall et al. [37] and Hachicha et al. [38] also showed that increase in pH may occur as a result of the mineralization of OM and the decomposition of acids containing carboxylic and phenolic groups. After the observed increase, during days 28 to 42 of the maturation phase, the pH of the mixture stabilized at approximately 7 (neutral). Zenjari et al. [39] and El Fels et al. [24] showed that the stabilization of pH at near neutral values during composting results from the generation of humic substances, which act to increase buffering capacity. During the final week of the maturation phase (days 42 to 49), the pH decreased to 6.9, which remained constant at the end of composting; this was the result of compost maturation under aerobic conditions, the generation of humic acids [38, 40], and H⁺ release as a result of nitrification by nitrifying bacteria during the later stages of composting [33]. The final pH values of the composts were 6.9, which matches the recommended pH for matured compost products [41].

3.2.2. EC variation

EC indicates the degree of compost salinity and the quantity of ions in the final composting product; this parameters reflect the potential product phytotoxicity. EC increased sharply from 1.7 mS cm⁻¹ to 3.1 mS cm⁻¹ (Fig. 3). Huang et al. [32] showed that an increase in EC could be due to the release of mineral salts, such as phosphate and ammonium ions, occasioned by the degradation of organic materials. Additionally, concentration effects resulting from OM loss and water evaporation due to high temperatures can increase EC [42].

A similar EC increase observed by Zhang et al. [43] was attributed to water evaporation. The increased content of soluble salts indicated the progressive mineralization of OM [42]. By the end of the composting process, the EC values of the final compost were 3.1 ms cm⁻¹, below 4 ms cm⁻¹ which is commonly regarded as the limit for safe use on plants [44]. This indicates that the compost is safe for most crop growth. However, due to the lack of a definite pattern in the changes in pH and EC over time, these parameters were not considered suitable for monitoring the com-

posting process or for evaluating the stability of the composting material [45].

3.3. OM Loss and Ash Content

Approximately 50% of OM is fully mineralized [46]. The degradation of OM during composting can be estimated based on the loss of OM, and here, OM_{loss} increased sharply to 52.5% within the first 35 d of composting (Fig. 4) due to the greater availability of substances which are easily biodegradable by microorganisms. It then slowed down, reaching 56.7% at the end of composting due to the depletion of easily biodegradable carbon and the synthesis reactions of the new complex polymerized organic substances (humification), processes which prevail over mineralization during the maturation stage [47]. This transformation matched well with the remarkable reduction in OM, indicating a rapid decomposition rate. Expected OM loss during composting is between 30% and 60% and OM loss > 42% may be accepted as an index value for mature compost [48]. Based on this, the compost could be considered matured during 28 d. Composting is an exothermic aerobic process in which the OM in SS are decomposed and mineralized to CO₂ by microorganisms. As a result, a reduction in OM is always observed in SS composting. The higher OMloss observed in this case could be due to the presence of two thermophilic temperature peaks and the longer thermophilic periods experienced during composting using this new composting technology (Fig. 2). It has previously been found that high composting temperatures accelerate the growth of thermophilic microorganisms, allowing further biodegradation of OM [49].

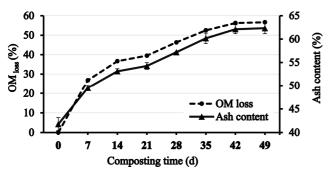


Fig. 4. Evolution of OMloss and ash content during composting.

During composting, there was an increase in ash content in the windrow. The ash content increased by 62.3% after 49 d (Fig. 4). Several authors have stated that increased ash content during composting results from the mineralization of OM by microbes [50, 51], and this parameter indicates decomposition and mineralization of the OM, consequently reflecting the degree of stabilization achieved during the process. Final compost with a higher ash content and lower OM is considered mature.

3.4. C/N Ratio

The C/N ratio is one of the most significant parameters used to evaluate the stabilization of compost. In this case, the C/N ratio decreased to reach a value of approximately 6.7 (Fig. 5) after 49 d of composting. The continuous and rapid reduction in the C/N

ratio was due to loss of TC and an increase in the proportion of TN due to the concentration effects resulting from the intense biological oxidation of OM over the composting period [32]. The TN value might have been also increased within the compost under the GORE(R) cover membrane due to the contribution of the free-living N-fixing microorganisms that are generated during later stages of the composting process [30]. Several authors have shown no agreement regarding the best C/N ratio for stable compost. However, Chefetz et al. [52], Provenzano et al. [53], and El Fels et al. [24] all state that values near to 10 indicate low microbial activity and result in a stabilized compost. According to Tomati et al. [54], a C/N ratio of between 11 and 22 indicates stabilized compost. In the present study, the C/N ratio of the final compost reached a value of 6.7, which indicated that compost stabilization was achieved. Using this technology, the C/N ratio reduction rate was higher than the C/N ratios achieved in conventional methods of composting with the same substrate, in which C/N ratios were found reduce to 10.1 [24], 11.7 [48], and 12.9 [55]. This is due to the forced bottom-up aeration system, which improves aeration conditions, accelerates decomposition, and results in a greater loss of carbon as carbon dioxide. The results thus agree with the findings of Guo et al. [18], who demonstrated that higher rates of aeration lead to an enhancement of carbon loss.

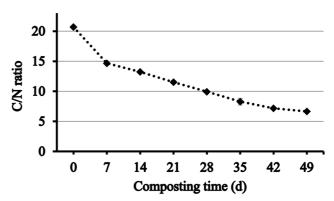


Fig. 5. Evolution of the C/N ratio during composting.

3.5. NH₄+/NO₃- Ratio

The NH₄⁺/NO₃ ratio ranged from 20.3 to 0.1 after 49 d of composting (Fig. 6). The NH₄⁺/NO₃ ratio is used to indicate the maturity of compost; a ratio below the limit of 0.16 for composting materials such as SS, municipal solid waste, and pig slurry indicates maturity [47]. The recorded value of approximately 0.1 indicated that the final product reached maturity in this case, and that the substrate had been changed to yield non-phytotoxic compost. Garcia et al. [56] and El Fels et al. [24] showed that a ratio below 1.0 suggests that the final compost has reached maturity. For comparison, the critical limits for maturity during composting of SS by conventional composting technology have been found to be fulfilled after 6 mon [24], 220 d [57], and 135 d [48], while the new composting technology achieved maturity critical limits by the 49th d, implying that the conventional composting technology takes much longer to achieve the index value for maturity than the new composting technology, most likely due to the differences in aeration. These results indicate that this new technology is highly suitable for ensuring compost maturity in a shorter time frame due to improved aeration conditions and the flow oxygen throughout the entire composting vessel using forced bottom-up aeration that enhance the oxidization processes, leading to an increase in the intensity of nitrification [58].

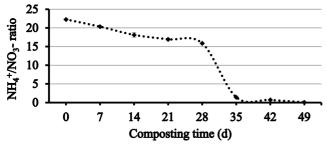


Fig. 6. Evolution of the NH4+/NO3- ratio during composting.

3.6. CEC Evolution

During composting, CEC increased substantially from 51.3 cmol kg-1 to 170.3 cmol kg-1 after 49 d (Fig. 7). CEC can be used to evaluate the degree of humification and the nutrient retention capacity of compost [59, 60]. According to Harada and Inoko [20], the CEC of a mature compost should be > 60 cmol kg⁻¹. In the current study, the CEC value of the final product was 170.3 cmol kg⁻¹, which indicates that the compost is mature. Zhang and Sun [60] confirmed that high CEC values are due to larger proportions of carboxyl functional groups in the substrates, while Lax et al. [61] concluded that increases in CEC values during composting could be the result of the accumulation of compounds bearing a negative charge, such as lignin-derived products, carboxyl groups, and phenolic hydroxyl groups. Joseph et al. [62] further showed that the presence of negatively charged groups caused the attraction, retention, and exchange of basic cations, which in turn could increase the CEC and nutrient-holding capacity of the compost. Any increase in CEC during composting is due to the humification process, during which carboxyl and phenolic functional groups are produced [47]. Humic compounds have high capacity to adsorb positively charged ions, which are then easily exchanged with other cations at the same adsorption sites [41].

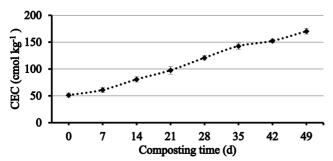


Fig. 7. CEC evolution during composting.

3.7. UV-visible Spectroscopic Analysis

The evolution of the $E_{2/6}$, $E_{2/4}$, and $E_{4/6}$ ratios provides information on the degree of OM maturity during composting. The E_{2/4}, E_{2/6} and E_{4/6} ratios decreased sharply to 5.1, 25.4, and 3.8, respectively, after 49 d of composting (Table S1). These decreases indicate the occurrence of the humification process. The decreases in the $E_{2/6}$ and E_{4/6} ratios at the end of composting demonstrated a strong increase in the humification of OM [55]. Chen et al. [63] used the $E_{4/6}$ ratio as a humification index, and Zbytniewski and Buszewski [22] reported that low $E_{2/6}$ or $E_{4/6}$ ratios reflected high degrees of aromatic condensation and indicated a higher level of organic material humification. E4/6 can also be used as an estimator of molecular weight [63]. Guo et al. [64] showed that during the thermophilic stage, rapid breakdown of lignin and quinine moieties occurred as the E2/4 ratio decreased, indicating a higher degree of aromatic condensation, as well as a higher level of organic material humification. Therefore, a continuous decrease in $E_{2/4}$ reflects a high degree of aromatic condensation.

3.8. FTIR Spectroscopic Analysis

FTIR spectrometry is a non-destructive qualitative or finger printing method used to characterize the principal classes of chemical groups that make up OM [65], in order to determine the changes seen in OM during composting [66]. The FTIR spectra thus derived are presented in Fig. S1. A very wide band located at 3,460 cm⁻¹ was detected during the process that can be attributed to the stretching vibrations of OH in hydroxyl groups. El Mezouari El Glauoi et al. [66] attributed this band to the hydrogenic vibrations of OH groups of alcohols and to phenols or to the OH of the carboxyl groups (COOH). The interaction of hydroxyl groups with hydrogen bonds explains the wide profile of this band and the fact that the wave number is lower than that of a free OH hydroxyl [24]. El Fels et al. [67] reported that the 3,000-2,800 cm⁻¹ region reflects the hydrophobic properties of aliphatic OM. The peak observed at 2,950 cm⁻¹ is due to methyl derived from methylene C-H aliphatic groups. The intensity of these bands decreases during composting, which is a good indication of the microbial oxidation of OM, especially the aliphatic compounds, during composting [24]. The peak intensity ratio of the bands at 2,920 and 1,640 cm⁻¹ (designated as aliphatic methylene and aromatic C, respectively) is suggested as a meaningful parameter for tracking the biological stability of OM during landfill stabilization [68, 69].

The band located at approximately 1,675 cm⁻¹ showed an increase during composting. El Fels et al. [24] attributed the region from 1,750-1,700 cm⁻¹ to vC = O stretching of the carboxyl group in its nonionized form, and the peaks that appear at approximately 1,650 cm⁻¹ to vC = O of ionized carboxyl (COO-). According to Kaiser and Ellerbrock [70], the band at 1,650 cm⁻¹ is associated with the vibrations of vC = C aromatic structures and the antisymmetric vibrations of COO-. In a biological specimen study, Maquelin et al. [71] further attributed the 1,675 cm⁻¹ band to the amide I band components resulting from the antiparallel pleated sheets and β -turns of proteins. Pajączkowska et al. [72] observed absorbance bands between 1,680 and 1,630 cm⁻¹ and attributed these to vCOO- in the case of humic acids extracted from sludge at different stages of treatment. An increase in this band thus pro-

vides information on the evolution of humification of OM during composting. Smidt et al. [73] showed that humic acids display a strong band at 1,640 cm⁻¹ that increases in intensity during the composting process. The continuous increase in the humic acid content has a positive impact on this band. The bands at 1,470 cm⁻¹ are attributed to aromatic benzene compounds. Droussi et al. [74] attributed the region from 1,442-1,472 to C = C stretching vibrations arising from aromatic components. An increase in the intensity of the aromatic absorbance indicates progress in the humification process [24, 66]. The 1,170 cm⁻¹ band can be attributed to C-O vibrations. While analysing fulvic acids, Zhou et al. [75] attributed the band at 1,170 cm⁻¹ to C-O stretching in alcohols, and El Fels et al. [24] reported that the change in intensity of the observed bands at 1,740 cm⁻¹ and 1,155 cm⁻¹ indicated the presence of a carboxyl functional group in the first, while that seen in the second is probably a characteristic of an ester bond. The 1,160 cm⁻¹ band is assigned to the asymmetric stretching of C-O-C of glycosidic linkages [76]. Ouatmane et al. [77] and Wu et al. [69] attributed the 1,165 cm⁻¹ band to C-O-C stretching; El Mezouari El Glauoi et al. [66] showed that the latter band could also be used as an indicator of the biodegradation process. The 1,067 cm⁻¹ band remained constant during composting. The region at 1,080-1,030 cm⁻¹ was attributed to the C-O stretching of polysaccharides or polysaccharide-like substances and the Si-O asymmetric stretch of silicate impurities [78, 79]. Aguelmous et al. [80] observed bands located at 875 and 713 cm⁻¹, which suggest the presence of calcium carbonates. The band at 875 cm⁻¹ could be attributed to the C-O out of plane vibration of carbonates. Reig et al. [81] attributed the 875 cm⁻¹ band to the C-O out-of-plane bending of carbonate. The evolution of each functional group reflects the advanced stage of humification of OM during composting.

3.9. Characteristics of the Final Compost

Compost product can be safely used as organic fertilizer or conditioner once it is mature or stable and does not present toxicity to plant growth [41]. Table S2 shows the main chemical properties of the final composts obtained in this experiment. The final compost product satisfied the threshold levels established in the literature for common maturation indices, suggesting that it can be used as an organic fertilizer for plant growth. These findings indicated that composting was carried out successfully under optimized conditions.

4. Conclusions

The current study indicates that ELS composting technology is an effective method for the disposal of SS waste based on the production of mature compost. The final compost product met the threshold levels established in the literature for the subsequent use of such compost as a soil amendment or as organic fertiliser for soil based on it obtaining the following parameters: pH 7.1-7.7, EC < 4 mS cm $^{-1}$, C/N ratio < 10, NH $_4$ +/NO $_3$ - < 0.16, CEC > 60 cmol kg $^{-1}$, and OM 33.3-55.4%. This new technology is novel and is extremely effective due to its two thermophilic temperature peaks, as well as rapid onset, and long-duration thermophilic phase. Moreover, the proposed new technology shortens SS composting time to obtain mature compost in only 49 d, unlike conventional

composting technology that requires 105-180 d and produces more immature compost. Further studies are recommended to focus on the evolution of microbial communities and enzymatic activities in this new process.

Acknowledgments

This study was conducted with the financial support of the Stipendium Hungaricum Scholarship and the Tempus Public Foundation. I would also like to offer deep thanks to Eng. Zoltan Bernath and Eng. Padra István from the BÁCSVÍZ for their assistance in the successful completion of the work.

References

- Banegas V, Moreno JL, Moreno JI, García C, León G, Hernández T. Composting anaerobic and aerobic sewage sludges using two proportions of sawdust. Waste Manage. 2007;27:1317-1327.
- Neyens E. Advanced sludge treatment affects extracellular polymeric substances to improve activated sludge dewatering. J. Hazard. Mater. 2004;106:83-92.
- 3. Leonard A. Management of wastewater sludge's: A hot topic at the European level. *J. Environ. Stud.* 2011;8:38.
- Senesi N, Plaza C, Brunetti G, Polo A. A comparative survey of recent results on humic-like fractions in organic amendments and effects on native soil humic substances. Soil Biol. Biochem. 2007;39:1244-1262.
- Cesaro A, Belgiorno V, Guida M. Compost from organic solid waste: Quality assessment and European regulations for its sustainable use. Resour. Conserv. Recycl. 2015;94:72-79.
- Zhou Y, Selvam A, Wong JWC. Chinese medicinal herbal residues as a bulking agent for food waste composting. *Bioresour. Technol.* 2018;249:182-188.
- Lim LY, Bong CPC, Lee CT, Klemeš JJ, Sarmidi MR, Lim JS. Review on the current composting practices and the potential of improvement using two-stage composting. *Chem. Eng. Trans.* 2017;61:1051-1056.
- Kulikowska D, Klimiuk E. Organic matter transformations and kinetics during sewage sludge composting in a two-stage system. *Bioresour. Technol.* 2011;102:10951-10958.
- Zhang L, Sun X, Tian Y, Gong X. Effects of brown sugar and calcium superphosphate on the secondary fermentation of green waste. *Bioresour. Technol.* 2013;131:68-75.
- Khalil A, Domeizel M, Prudent P. Monitoring of green waste composting process based on redox potential. *Bioresour. Technol.* 2008:99:6037-6045.
- González I, Robledo-Mahón T, Silva-Castro GA, et al. Evolution of the composting process with semi-permeable film technology at industrial scale. J. CLEAN Prod. 2016;115:245-254.
- Robledo-Mahón T, Aranda E, Pesciaroli C, et al. Effect of semi-permeable cover system on the bacterial diversity during sewage sludge composting. J. Environ. Manage. 2018;215:57-67.
- 13. Ma S, Fang C, Sun X, Han L, He X, Huang G. Bacterial community succession during pig manure and wheat straw aerobic composting covered with a semi-permeable membrane under slight

- positive pressure. Bioresour. Technol. 2018;259:221-227.
- 14. Ma S, Sun X, Fang C, He X, Han L, Huang G. Exploring the mechanisms of decreased methane during pig manure and wheat straw aerobic composting covered with a semi-permeable membrane. Waste Manage. 2018;78:393-400.
- Sun X, Ma S, Han L, et al. The effect of a semi-permeable membrane-covered composting system on greenhouse gas and ammonia emissions in the Tibetan Plateau. J. CLEAN Prod. 2018;204:778-787.
- Mukhopadhyay A, Midha VK. A review on designing the waterproof breathable fabrics. Part II: Construction and suitability of breathable fabrics for different uses. J. Ind. Text. 2008;38:17-41.
- 17. TMECC. Test methods for the examination of composting and compost. Thompson WH, Leege PB, Millner PD, Watson ME, eds. The United States Composting Council Research and Education Foundation. The United States Department of Agriculture: 2002.
- Guo R, Li G, Jiang T, et al. Effect of aeration rate, C/N ratio and moisture content on the stability and maturity of compost. *Bioresour. Technol.* 2012;112:171-178.
- Paredes C, Roig A, Bernal MP, Sánchez-Monedero MA, Cegarra J. Evolution of organic matter and nitrogen during co-composting of olive mill wastewater with solid organic wastes. *Biol. Fertil.* Soil. 2000;32:222-227.
- Harada Y, Inoko A. Relationship between cation-exchange capacity and degree of maturity of city refuse composts. Soil Sci. Plant Nutr. 1980;26:353-362.
- Okalebo JR, Gathua KW, Woomer PL. Laboratory methods of soil and plant analysis: A working manual. 2nd ed. Nairobi, Kenya: Sacred African Publishers; 2002.
- 22. Zbytniewski R, Buszewski B. Characterization of natural organic matter (NOM) derived from sewage sludge compost. Part 1: Chemical and spectroscopic properties. *Bioresour. Technol.* 2005;96:471-478.
- Amir S, Hafidi M, Merlina G, Revel J-C. Structural characterization of fulvic acids during composting of sewage sludge. *Process Biochem.* 2005;40:1693-1700.
- 24. El Fels L, Zamama M, El Asli A, Hafidi M. Assessment of biotransformation of organic matter during co-composting of sewage sludge-lignocelullosic waste by chemical, FTIR analyses, and phytotoxicity tests. *Int. Biodeterior. Biodegrad.* 2014;87: 128-137.
- 25. Insam H, Franke-Whittle I, Goberna M. Microbes in aerobic and anaerobic waste treatment. In: Microbes at work. Berlin, Heidelberg: Springer Berlin Heidelberg; 2010;353:1-34.
- Zhang L, Sun X. Effects of bean dregs and crab shell powder additives on the composting of green waste. *Bioresour. Technol.* 2018;260:283-293.
- 27. Zhang L, Sun X. Influence of sugar beet pulp and paper waste as bulking agents on physical, chemical, and microbial properties during green waste composting. *Bioresour. Technol.* 2018;267:182-191.
- Zhang L, Sun X. Evaluation of maifanite and silage as amendments for green waste composting. Waste Manage. 2018;77: 435-446.
- 29. Tønner-Klank L, Møller J, Forslund A, Dalsgaard A. Microbiological assessments of compost toilets: In situ measure-

- ments and laboratory studies on the survival of fecal microbial indicators using sentinel chambers. *Waste Manage*. 2007;27: 1144-1154.
- 30. de Bertoldi M, Vallini G, Pera A. The biology of composting: A review. *Waste Manage. Res.* 1983;1:157-176.
- El Hayany B, El Glaoui GEM, Rihanni M, et al. Effect of dewatering and composting on helminth eggs removal from lagooning sludge under semi-arid climate. *Environ. Sci. Pollut. Res.* 2018;25:10988-10996.
- 32. Huang GF, Wong JWC, Wu QT, Nagar BB. Effect of C/N on composting of pig manure with sawdust. *Waste Manage*. 2004;24:805-813.
- Eklind Y, Kirchmann H. Composting and storage of organic household waste with different litter amendments. II: Nitrogen turnover and losses. *Bioresour. Technol.* 2000;74:125-133.
- Cáceres R, Malińska K, Marfà O. Nitrification within composting: A review. Waste Manage. 2018;72:119-137.
- Barrington S, Choiniere D, Maher T, Knight W. Effect of carbon source on compost nitrogen and carbon losses. *Bioresour. Technol.* 2002;83:189-194.
- 36. Vergnoux A, Guiliano M, Le Dréau Y, Kister J, Dupuy N, Doumenq P. Monitoring of the evolution of an industrial compost and prediction of some compost properties by NIR spectroscopy. Sci. Total Environ. 2009;407:2390-2403.
- 37. Baeta-Hall L, Céu Sàágua M, Lourdes Bartolomeu M, Anselmo AM, Fernanda Rosa M. Bio-degradation of olive oil husks in composting aerated piles. *Bioresour. Technol.* 2005;96:69-78.
- 38. Hachicha S, Sallemi F, Medhioub K, Hachicha R, Ammar E. Quality assessment of composts prepared with olive mill wastewater and agricultural wastes. *Waste Manage*. 2008;28:2593-2603.
- 39. Zenjari B, El Hajjouji H, Ait Baddi G, et al. Eliminating toxic compounds by composting olive mill wastewater-straw mixtures. *J. Hazard. Mater.* 2006;138:433-437.
- Michailides M, Christou G, Akratos CS, Tekerlekopoulou AG, Vayenas DV. Composting of olive leaves and pomace from a three-phase olive mill plant. *Int. Biodeterior. Biodegrad.* 2011;65:560-564.
- 41. Azim K, Soudi B, Boukhari S, Perissol C, Roussos S, Thami Alami I. Composting parameters and compost quality: A literature review. *Org. Agric.* 2018;8:141-158.
- 42. Cáceres R, Flotats X, Marfà O. Changes in the chemical and physicochemical properties of the solid fraction of cattle slurry during composting using different aeration strategies. Waste Manage. 2006;26:1081-1091.
- Zhang D, Luo W, Li Y, Wang G, Li G. Performance of co-composting sewage sludge and organic fraction of municipal solid waste at different proportions. *Bioresour. Technol.* 2018;250:853-859.
- 44. Singh WR, Kalamdhad AS. Potential for composting of green phumdi biomass of Loktak lake. *Ecol. Eng.* 2014;67:119-126.
- 45. Khan MAI, Ueno K, Horimoto S, Komai F, Tanaka K, Ono Y. Physicochemical, including spectroscopic, and biological analyses during composting of green tea waste and rice bran. *Biol. Fertil. Soil.* 2009;45:305-313.
- 46. Bernal MP, Sommer SG, Chadwick D, Qing C, Guoxue L, Michel FC. Current approaches and future trends in compost quality criteria for agronomic, environmental, and human health benefits. In: Advances in agronomy. 2017;144:143-233.

- Bernal MP, Alburquerque JA, Moral R. Composting of animal manures and chemical criteria for compost maturity assessment. A review. *Bioresour. Technol.* 2009;100:5444-5453.
- Raj D, Antil RS. Evaluation of maturity and stability parameters of composts prepared from agro-industrial wastes. *Bioresour. Technol.* 2011;102:2868-2873.
- 49. Meng L, Li W, Zhang S, Wu C, Lv L. Feasibility of co-composting of sewage sludge, spent mushroom substrate and wheat straw. *Bioresour. Technol.* 2017;226:39-45.
- Tognetti C, Mazzarino MJ, Laos F. Improving the quality of municipal organic waste compost. *Bioresour. Technol.* 2007;98:1067-1076.
- Rihani M, Malamis D, Bihaoui B, Etahiri S, Loizidou M, Assobhei O. In-vessel treatment of urban primary sludge by aerobic composting. *Bioresour. Technol.* 2010;101:5988-5995.
- Chefetz B, Hatcher PG, Hadar Y, Chen Y. Chemical and biological characterization of organic matter during composting of municipal solid waste. *J. Environ. Qual.* 1996;25:776-785.
- 53. Provenzano MR, de Oliveira SC, Santiago Silva MR, Senesi N. Assessment of maturity degree of composts from domestic solid wastes by fluorescence and fourier transform infrared spectroscopies. J. Agric. Food Chem. 2001;49:5874-5879.
- Tomati U, Belardinelli M, Andreu M, et al. Evaluation of commercial compost quality. Waste Manage. Res. 2002;20:389-397.
- Lahsaini S, Aguelmous A, El Fels L, et al. Assessment of biotransformation of sludge from vegetable oil refining by composting. *Compost Sci. Util.* 2017;25:130-140.
- 56. García C, Hernández T, Costa F. Characterization of humic acids from uncomposted and composted sewage sludge by degradative and non-degradative techniques. *Bioresour. Technol.* 1992:41:53-57.
- 57. Jouraiphy A, Amir S, El Gharous M, Revel J-C, Hafidi M. Chemical and spectroscopic analysis of organic matter transformation during composting of sewage sludge and green plant waste. *Int. Biodeterior. Biodegrad.* 2005;56:101-108.
- 58. Hao X, Chang C. Gaseous NO, NO₂, NH₃ loss during cattle feedlot manure composting. *Phyton* 2001;41:81-94.
- 59. Roig A, Lax A, Cegarra J, Costa F, Hernandez MT. Cation exchange capacity as a parameter for measuring the humification degree of manures. Soil Sci. 1988;146:311-316.
- 60. Zhang L, Sun X. Changes in physical, chemical, and microbiological properties during the two-stage co-composting of green waste with spent mushroom compost and biochar. Bioresour. Technol. 2014;171:274-284.
- Lax A, Roig A, Costa F. A method for determining the cation-exchange capacity of organic materials. *Plant Soil* 1986;94:349-355.
- 62. Joseph SD, Camps-Arbestain M, Lin Y, et al. An investigation into the reactions of biochar in soil. Soil Res. 2010;48:501-515.
- Chen Y, Senesi N, Schnitzer M. Information provided on humic substances by E4/E6 ratios. Soil Sci. Soc. Am. J. 1977;41:352-358.
- 64. Guo X, Huang J, Lu Y, Shan G, Li Q. The influence of flue gas desulphurization gypsum additive on characteristics and evolution of humic substance during co-composting of dairy manure and sugarcane pressmud. *Bioresour. Technol.* 2016;219: 169-174.
- 65. Chen Y. Nuclear magnetic resonance, infra-red and pyrolysis: Application of spectroscopic methodologies to maturity deter-

- mination of composts. Compost Sci. Util. 2003;11:152-168.
- 66. El Mezouari El Glauoi G, El Hayany B, El Fels L, et al. Physico-chemical and spectroscopy assessment of sludge biodegradation during semi-industrial composting under semi-arid climate. Waste Biomass Valori. 2018:1-12.
- 67. El Fels L, Zamama M, Hafidi M. Advantages and limitations of using FTIR spectroscopy for assessing the maturity of sewage sludge and olive oil waste co-composts. In: Chamy R, Rosenkranz F, Soler L, eds. Biodegradation and bioremediation of polluted systems – New advances and technologies. InTech; 2015. p.127-144.
- 68. Lü F, Shao L-M, Zhang H, et al. Application of advanced techniques for the assessment of bio-stability of biowaste-derived residues: A mini review. *Bioresour. Technol.* 2018;248:122-133.
- 69. Wu H, Zhao Y, Long Y, Zhu Y, Wang H, Lu W. Evaluation of the biological stability of waste during landfill stabilization by thermogravimetric analysis and Fourier transform infrared spectroscopy. *Bioresour. Technol.* 2011;102:9403-9408.
- Kaiser M, Ellerbrock RH. Functional characterization of soil organic matter fractions different in solubility originating from a long-term field experiment. Geoderma 2005;127:196-206.
- Maquelin K, Kirschner C, Choo-Smith L-P, et al. Identification of medically relevant microorganisms by vibrational spectroscopy. J. Microbiol. Meth. 2002;51:255-271.
- Pajączkowska J, Sułkowska A, Sułkowski WW, Jędrzejczyk M. Spectroscopic study of the humification process during sewage sludge treatment. J. Mol. Struct. 2003;651-653:141-149.
- 73. Smidt E, Eckhardt K, Lechner P, Schulten H, Leinweber P. Characterization of different decomposition stages of biowaste using FT-IR spectroscopy and pyrolysis-field ionization mass spectrometry. *Biodegradation* 2005;16:67-79.
- 74. Droussi Z, D'orazio V, Provenzano MR, Hafidi M, Ouatmane A. Study of the biodegradation and transformation of olive-mill residues during composting using FTIR spectroscopy and differential scanning calorimetry. J. Hazard. Mater. 2009;164:1281-1285.
- Zhou Y, Selvam A, Wong JWC. Evaluation of humic substances during co-composting of food waste, sawdust and Chinese medicinal herbal residues. *Bioresour. Technol.* 2014;168:229-234.
- Bower DI, Maddams WF. The vibrational spectroscopy of polymers. Cambridge: Cambridge Univ. Press; 1989.
- 77. Ouatmane A, Provenzano MR, Hafidi M, Senesi N. Compost maturity assessment using calorimetry, spectroscopy and chemical analysis. *Compost Sci. Util.* 2000;8:124-134.
- Filip Z, Bielek P. Susceptibility of humic acids from soils with various contents of metals to microbial utilization and transformation. *Biol. Fertil. Soil.* 2002;36:426-433.
- Smidt E, Lechner P, Schwanninger M, Haberhauer G, Gerzabek MH. Characterization of waste organic matter by FT-IR spectroscopy: Application in waste science. *Appl. Spectrosc.* 2002;56: 1170-1175.
- Aguelmous A, Lahsaini S, El Fels L, Souabi S, Zamama M, Hafidi M. Assessment of organo-mineral fraction during storage of oil refinery sludge. *Moroccan J. Chem.* 2016;4:1029-1036.
- 81. Reig FB, Adelantado JVG, Moya Moreno MCM. FTIR quantitative analysis of calcium carbonate (calcite) and silica (quartz) mixtures using the constant ratio method. Application to geological samples. *Talanta* 2002;58:811-821.