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Application of differential scanning calorimetry (DSC) to evaluate the quality of compost organic matter

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Abstract

Differential scanning calorimetry (DSC) was used to describe compost organic matter quality with regard to input materials and humification. Several basic experiments using 10 selected compost samples were performed to find out how material composition and humification affect the heat flow of the sample and the shift of the second exothermic organic peak. Each input material contributes to the specific DSC profile of the whole compost sample. Biowaste composts from the separate collection can be distinguished from sewage sludge composts due to the specific thermal pattern. Despite the influence of the material itself on the thermal behavior, chemical changes of the progressing process effect the heat flow. Humification of biogenic waste materials causes increasing intensities of the second exothermic peak. Principal component analysis (PCA) was applied to reveal the inherent features in the DSC profiles of 24 compost samples (final products). Thermal characteristics enable the assignment of different materials (sewage sludge and biowaste) to their compost class according to the intended use. Based on these specific thermal features quality assessment of compost organic matter can be performed very fast. DSC profiles of humic acids extracted from the 10 selected composts were recorded. Although similarities of the thermal behavior are visible, each DSC profile shows individual properties with regard to peak position and intensity. Different carbon and nitrogen contents confirm the particular composition of compost humic acids.

Keywords: Differential scanning calorimetry (DSC); Humification; Compost; Composition

1. Introduction

Bioresource recovery is a keyword for current waste management strategies. Besides anaerobic digestion composting is a main recycling path and an appropriate way to treat organic waste materials, especially yard waste with lignocellulosic plant materials that are not degraded under anaerobic conditions. Composts are used as soil conditioners to close the biological cycle. Application in agriculture implies a separate collection of biowaste to ensure a better quality of the final product, and the compliance with the defined standard quality. Sewage sludge composts are used in landscaping. Due to the different fields of application differentiation of the input materials and improvement of compost quality are environmental and economic concerns. Waste management strategies aim to optimize mixtures with the right treatment (compost “design”) for each purpose to provide additional benefits. Up to now standard quality has focused primarily on the absence of pollutants. In the past little attention was paid to the quality of compost organic matter that is usually characterized by sum parameters such as loss of ignition, organic carbon and nitrogen. A more concise definition of organic matter quality could enhance the economic value of the product. To achieve this target in practice suitable parameters and adequate analytical tools that allow easy handling are necessary. Due to their favorable properties humic acids of compost organic matter are suggested as a “quality parameter”. Composting is the only way to build up humic substances in a technological process. Humification of biomolecules provides environmental benefits by carbon sequestration in that it reduces the CO_2 release caused by mineralization. Restitution of stable organic matter to soils is an environmental request facing the considerable loss of organic matter in agricultural soils [1]. In addition to the environmental aspect, benefits of humic substances for soils and plant health are well known [2]. With

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regard to improved humification, yard and kitchen waste are suitable substrates to be operated in aerobic processes (composting) as they provide aromatic moieties to build up humic substances. New strategies suggest the use of humic substances from waste as basic compounds for chemical syntheses, i.e. as surfactants [3,4]. Many attempts have been made to characterize soil humic substances including the soluble fractions of humic and fulvic acids and insoluble humins [5]. In waste management humified matter has been primarily considered as an indicator of organic matter stability [6]. Compost humic acids are "young" compared to soil humic acids. Despite ongoing transformation processes they represent a stable organic matter fraction. Their changes during composting and the long-term behavior of compost humic acids have been presented previously [7].

The application of diverse thermal methods for waste characterization, especially for the progress of stabilization during biological processes, has been described by several authors [8–11].

In the present study emphasis was placed on differential scanning calorimetry (DSC) to assess compost organic matter quality in terms of input materials and humification. The target was to find out how these compost properties are reflected by the DSC profile. Twenty-four biowaste and sewage sludge composts (final products) were characterized by means of their DSC profile and conventional parameters. Humic acid fractions from 10 selected composts were investigated by DSC in order to get more insight into their thermal behavior.

2. Materials and methods

2.1. Materials and sample preparation

Twenty-four compost samples (final products) originated from 20 different composting plants processing biowaste and/or sewage sludge. Ten composts were selected for further investigations of the isolated humic acid fraction. Criteria for selection were the differences of the input materials (sewage sludge or biowaste) and various humic acid contents. Table 1 compiles some information on input materials and process operation of the investigated composts. Biowaste comprises a mixture of yard and kitchen waste. The main compounds of sewage sludge composts are anaerobic digested sewage sludge, bulk material (yard waste or wooden compounds), and earth. Small amounts of leftovers from restaurants are allowed to be co-composted. Special ingredients except for the main components are roughly indicated in Table 1. Information on the ratio of ingredients and additional ingredients was not available. Several process parameters are missing as well (n.i. = no information). The compost samples (about 2 kg, sieved <10–15 mm) were provided by the company. Determination of nitrate and respiration activity was carried out from the fresh sample. For other analyses, samples were air-dried, ground with an agate mill and sieved <0.63 mm.

2.2. Chemical investigations and biological tests

The dry organic matter content (loss of ignition = LOI) was determined by combustion at 550 °C [12]. Total and inorganic carbon were analyzed by combustion using a Vario Max CNS Analyzer. Organic carbon was calculated by subtracting inorganic carbon from total carbon. Nitrogen of the compost samples was determined according to the Kjeldahl method, nitrate-N according to Austrian Standards [12]. Humic acids were extracted during 24 h using 0.1 molar Na-pyrophosphate solution (pH 10.5), and precipitated by means of 37% HCl. Humic acid contents are measured photometrically and calculated on the basis of gravimetric determinations. The extraction procedure is repeated four times as described by Gerzabek et al. [13]. Total carbon (TC) and total nitrogen (TN) in humic acids were determined by combustion using a Vario Max CNS Analyzer. Two replicates were performed.

For further investigations extracted and precipitated humic acids were dialyzed (exclusion size $M_W = 12{,}000$) using deionized water until Na-pyrophosphate was eliminated. Compost residues were obtained by repeated alkaline extraction until the solution was colorless and subsequent washing with deionized water to pH 7. For thermal analysis the residues were freeze-dried and ground with pestle and mortar.

The oxygen uptake (respiration activity) during 4 days was determined in a sapromat (Sulzer Voith) as described by Binner and Zach [14].

2.3. Differential scanning calorimetry (DSC)

Thermal analysis was conducted in a STA 409 CD Skimmer (Netzsch GmbH) where the mass loss (thermogravimetry, TG) and DSC profiles are recorded simultaneously. These investigations focused on differential scanning calorimetry. Combustion took place under oxidative conditions within a temperature range from 30 to 950 °C, using a gas flow of 120 ml min^{-1} (20% O_2/80% He) and a constant heating rate of 10 K min^{-1}. The sample (16 mg) was combusted in an Al_2O_3 pan. Self-controlled calibration was carried out. DSC curves were corrected by subtracting the DSC curve of the empty pan from the recorded sample curve.

Enthalpies were calculated by integration of the area below the DSC curve between 30 and 625 °C, drawing a horizontal baseline (heat flow 0) from 30 to 625 °C (Proteus software tool).

DSC profiles of 10 replicates were recorded to proof the reproducibility. An average conformity of 99.1% was found by the OPUS software (range of 98.8–99.5% if each DSC profile is compared to the average).

2.4. Multivariate data analysis

Principal component analysis (PCA) and partial least squares regression (PLS-R) of DSC data were performed using the Unscrambler 9.2 (Camo). By means of PCA huge data pools are reduced in that data are decomposed into the abstract matrices of scores and loadings. Scores indicate the location of samples along each model component. Loadings show the contribution of variables to the model components. PCA provides an insight into the data structure and the underlying features. The number of components reveals the inherent dimensionality of the data [15,16].

Table 1
Input materials and process parameters: aeration (+ positive pressure or − negative pressure), maximum temperatures reached, process duration

Sample	Input materials	Forced aeration + or − pressure	T_{max} (°C)	Process (weeks)
P1	Yard waste	No	70	44
P2	Biowaste	+ pressure	70	37
P3	Biowaste	n.i.	n.i.	n.i.
P4	Biowaste, earth	+ pressure	75	23
P5	Sewage sludge, yard waste, earth	No	78	31
P6	Sewage sludge, paper slurry, charred grape marc	No	66	67
P7	Sewage sludge, yard waste	No	70	61
P8	Biowaste, leftovers	No	70	50
P9	Biowaste, leftovers	− pressure	70	38
P10	Biowaste, leftovers, earth	+ pressure	75	n.i.
P11	Biowaste, leftovers	+ pressure	n.i.	16
P12	Biowaste	+ pressure	70	41
P13	Sewage sludge, yard waste	− pressure	45	70
P14	Yard waste (biowaste)	n.i.	n.i..	n.i..
P15	Biowaste, yard waste	No	70	30
P16	Biowaste	+ pressure	85	20
P17	Sewage sludge, yard waste	+ pressure/no	65	17
P18	Sewage sludge, yard waste	− pressure	65	40
P19	Biowaste, yard waste	n.i.	n.i.	n.i.
P20	Sewage sludge, yard waste	− pressure	70	n.i.
P21	Biowaste	+ pressure	70	36
P22	Sewage sludge, yard waste, earth	No	70	25
P23	Sewage sludge, yard waste	No	65	60
P24	biowaste	No	70	61

3. Results and discussion

The obtained yield of extracted humic acids is very different in the composts depending on input materials and process operation. In well-humified composts extractable humic acid contents represent more than 30% of organic dry matter (ODM). Table 2 displays humic acid contents, and several data characterizing the investigated compost samples. Due to differences in process operation and plant capacities the composting period differs considerably. However, all composts investigated are mature with

Table 2
Humic acid (HA) contents, organic dry matter contents (loss of ignition = LOI), TOC, N_{kjel}, NO_3–N, C/N ratio, and respiration activities within 4 days (RA_4) of 24 composts originating from 20 different composting plants

Sample	HA (% ODM)	LOI (% DM)	TOC (% DM)	N_{kjel} (% DM)	NO_3–N (ppm)	C/N	RA_4 (mg O_2 g^{-1} DM)
P1	27.2	34.2	20.0	1.51	175	13	2.6
P2	32.0	37.9	20.5	2.41	880	9	1.9
P3	36.7	42.0	22.9	2.84	1050	8	3.7
P4	26.6	19.3	9.8	0.94	250	10	1.0
P5	13.2	21.7	10.7	1.04	1150	10	2.6
P6	8.9	41.3	21.0	1.81	420	12	2.6
P7	19.7	27.3	13.0	1.58	1700	8	2.6
P8	41.5	31.0	16.5	1.77	650	9	2.1
P9	45.6	28.8	14.9	1.54	800	10	2.3
P10	31.3	21.2	10.8	1.12	220	10	1.2
P11	31.4	39.7	21.5	2.31	25	9	2.1
P12	30.2	37.6	20.4	1.78	350	11	2.7
P13	26.6	23.8	11.4	1.15	1500	10	1.0
P14	29.2	47.5	26.6	1.97	175	14	5.0
P15	29.0	35.2	18.7	1.51	75	12	4.2
P16	25.7	44.9	24.4	2.36	125	10	4.4
P17	22.5	29.5	15.7	1.44	550	11	1.3
P18	15.1	23.9	11.9	1.16	900	10	2.5
P19	27.5	36.6	19.4	1.72	198	11	0.4
P20	19.6	35.8	19.3	1.60	210	12	4.2
P21	29.8	38.2	20.0	2.10	630	10	0.9
P22	21.4	19.1	9.4	0.94	170	10	0.9
P23	15.4	40.0	17.8	1.88	2300	9	2.0
P24	46.8	31.3	16.5	1.77	500	9	2.3

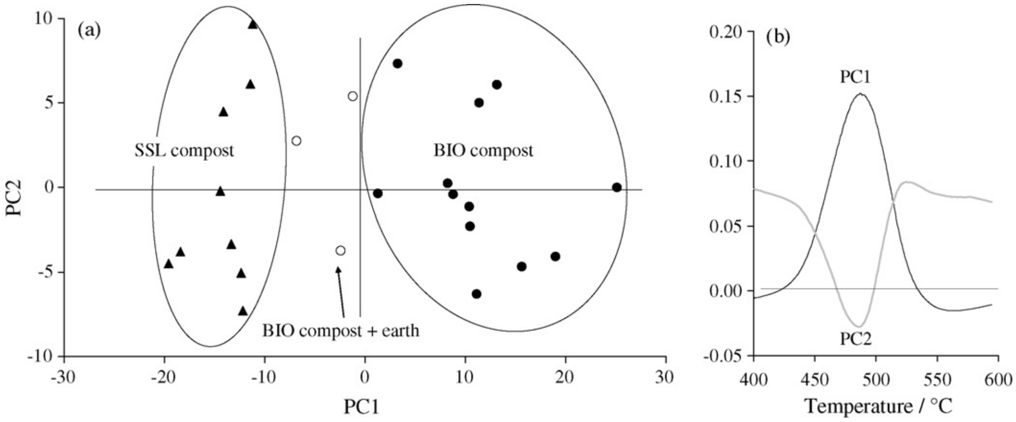

Fig. 1. PCA of 24 composts (scores plot) revealing two main classes of composts: sewage sludge (SSL) and biowaste (BIO) composts (a) and loadings of the first two PCs (b).

respect to conventional parameters such as respiration activity within 4 days (RA_4) and C/N ratios. Respiration activities $<7\,mg\,O_2\,g^{-1}$ DM and C/N ratios of 10–12 are considered significant for stable composts with the given composition.

Humic acid contents vary in a wide range. Smaller contents are usually found in sewage sludge composts (Table 2). A mixture that provides a diversity of chemical compounds with different degradability and more aromatic moieties fits better for humic substance synthesis. Biowaste composts meet in general these requirements. No correlation was found between humic acid contents and duration of composting and aeration systems. It can be concluded that the input material has more influence provided that process operation is performed properly. Longer storage of the final compost can slightly increase the humic acid contents. The stability of the material parallels a low microbial activity. Therefore, the progress of humification takes only place over a longer period of time.

Compared to conventional parameters it is evident that humic acid contents are independent of organic matter contents (LOI). A correlation of $R^2 = 0.09$ ($P = 0.7$) between humic acid and organic matter contents confirms this independency.

A PCA of DSC profiles was performed using the heat flow between 394 and 589 °C and four principal components (PC). This temperature range provided the highest discrimination power to separate the waste materials. The loadings of this area, shown in Fig. 1b for the 1st and 2nd PC, reveal the highest influence on variance. The first two components explain 91% of the total variance (81% and 10%; 3rd and 4th PC: 8%, and 1%, respectively). Fig. 1a illustrates the clustering of the composts in the scores plot, based on their DSC profiles. The 1st component discriminates sewage sludge and biowaste composts according to their main ingredients. Biowaste compost shows a prominent second exothermic peak that is missing in sewage sludge compost. This fact also explains the influence of this region on the loadings (Fig. 1b). The specific thermal feature of these two compost classes allows their differentiation which is important due to their divergent intended application. Biowaste with a high content of earth lies between biowaste and sewage sludge composts. Differences within each group are revealed by the 2nd PC. The widely spread arrangement of the samples in each compost group underlines the diversity of input materials within the group of biowaste or sewage sludge composts.

Despite the influence of the input material on the heat flow humification additionally affects the DSC profile considerably. The correlation between humic acid contents of the 24 composts and their heat flow, calculated by means of a partial least squares regression (PLS-R) resulted in $R^2 = 0.86$ at a significance level of 5%. The designated temperature region in Fig. 1b (loadings) was also responsible for this correlation. Although other underlying effects are included the contribution of humification is evident. To visualize the effect of humic acids on the DSC profile additional investigations were carried out using 10 selected composts representing sewage sludge and biowaste composts with different humic acid contents.

3.1. Thermoanalytical characterization of 10 selected composts and their corresponding humic acids

For detailed investigations 10 composts (P1–P10) that reflect the variety of components and mixtures were picked out. Fig. 2 shows changes of the heat flow (DSC profile) in the initial sample and the final sample (280 days) of compost P9 holding the highest humic acid content of the selected composts.

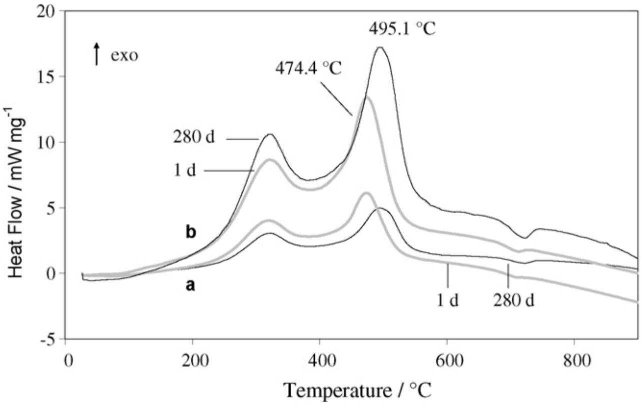

Fig. 2. DSC profiles of the initial biogenic waste (P9: 1 day) and the final compost (P9: 280 days) referring to dry matter (a) and organic dry matter (b).

Heat flow profiles of the input material and the final compost display a shift of the exothermic peak from 474.4 to 495.1 °C. The shift parallels the progressing maturation process that includes mineralization, humification, and enrichment of recalcitrant components. This effect has been described by several authors [8–10]. The intensity of the two exothermic peaks decreases during degradation and mineralization. Organic matter decomposition causes a decrease of enthalpy in the compost sample. However, referring to organic matter an increase of the exothermic peaks, especially of the second ones, is observed. Due to transformation of organic compounds an increase of the heat flow is recorded. Such transformation of organic matter can be traced back to humification. Melis and Castaldi reported on an increasing second exothermic peak in composts assigned to aromatic structures [10]. The evolution of more aromatic structures in mature composts due to the increase of humic acids was also investigated by Zaccheo et al. [17] using ^{13}C NMR spectroscopy. To verify the assumption that humic acids contribute to the second exothermic peak, DSC profiles of the compost residues were recorded after alkaline extraction of the humic acid fraction. In all biowaste composts the second exothermic peak of the DSC profiles is not longer visible after extraction. Provenzano et al. [18] and Provenzano and Senesi [19] attributed this peak in the high-temperature region (at about 500 °C) to oxidation and polycondensation of the aromatic core of humic substances. The change of the DSC profile of sewage sludge composts after alkaline extraction is less conspicuous because of the missing prominent second exothermic peak in the original DSC profile. Fig. 3 shows the compost with the highest humic acid content P9 (a) and the corresponding residue after alkaline extraction (b). The DSC curve reveals a specific pattern of the remaining organic and inorganic residue. The loss of the second exothermic peak in the biowaste composts indicates the contribution of humic acids to this heat flow peak. In addition broadening of the first exothermic peak between 330 and 450 °C and a shift to higher temperatures is observed in all composts investigated. This fact argues for the breakdown of complex structures after extraction, resulting in molecule moieties with lower combustion temperatures after removal of the "humic acid" fraction. Moreover, the endothermic peak of carbonates >700 °C is not found any longer in the residues, which is confirmed by the missing mass loss in the TG curves (not shown). At this moment it is not clear how the carbonates are affected by the extraction of humic acids.

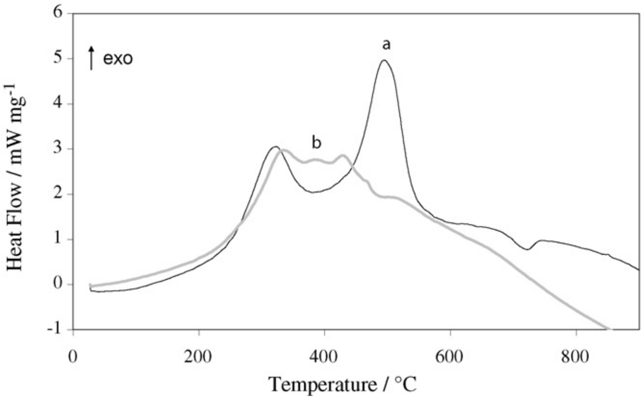

Fig. 3. DSC profiles of compost P9 (a) and of the residue after alkaline extraction of humic substances (b).

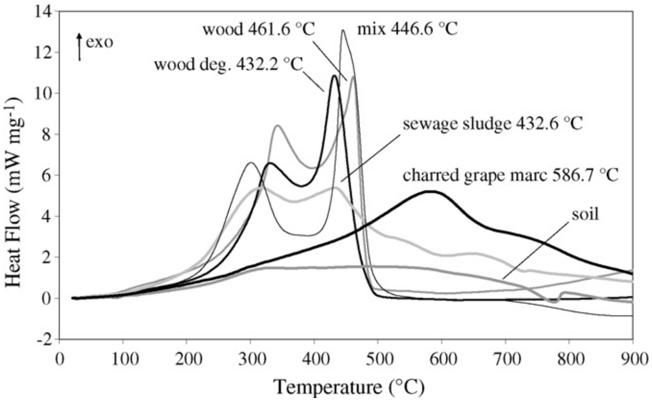

Fig. 4. Characteristic DSC profiles of compost ingredients: wood, degraded wood (wood deg.), mixture of fruits and vegetables (mix), sewage sludge, charred grape marc, and soil.

It can be assumed that besides other refractory organic matter fractions humic acids contribute to this second exothermic peak in the biowaste composts located between 480.5 °C (P4) and 497.1 °C (P3). Wooden compounds that are also enriched during the composting process show the peak maximum at lower temperatures (461.6–432.2 °C). Fig. 4 illustrates characteristic DSC profiles of wood and naturally degraded wood (wood deg.), a mixture of fruits and vegetables (mix), anaerobically digested sewage sludge from the waste water treatment plant, charred grape marc, and soil that are ingredients of the selected composts. All input materials have specific thermal features depending on the chemical compounds that influence the location of the exothermic peaks. In all input materials the second exothermic peak is found at lower temperatures than the peak of the mature composts. This finding indicates considerable changes by humification causing the second heat flow peak to shift to higher temperatures. Degradation of a singular component leads to a peak shift to lower temperatures (see degraded wood in Fig. 4). This fact suggests that a complex substrate is a prerequisite for humification processes, at least for their observation within the short period of composting. Soils show a characteristic flattened DSC curve compared to composts. Due to the close contact of the remaining resistant organic matter with mineral compounds the combustion process takes place moderately over a wide temperature range. A weak exothermic peak is observed between 300 and 400 °C as described by Salgado et al. [20]. Due to interactions that are not known in detail organic and mineral compounds influence the thermal behavior mutually.

The role of the input material becomes visible in the DSC curves of composts P5, P6, P7 that primarily contain sewage sludge. These composts have the lowest humic acid contents (13.2%, 8.9%, 19.7% referring to ODM) which is reflected by the missing second exothermic peak (Fig. 5). The special composition of compost P6 containing primarily proteins, lipids from sewage sludge and cellulose from paper sludge leads to the prominent first exothermic peak at 322 °C indicating combustion

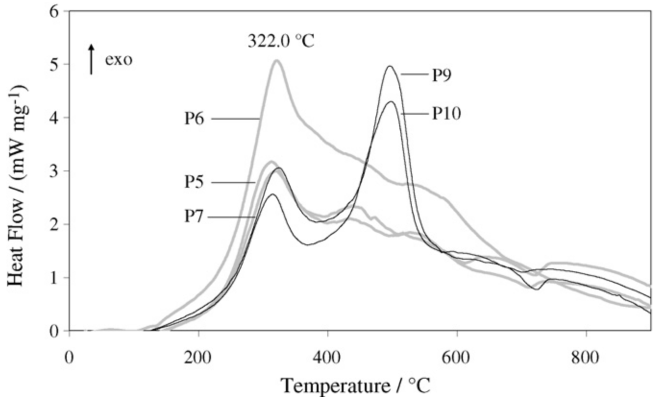

Fig. 5. DSC profiles of the biowaste composts P9 and P10 with HA contents of 45.6% and 31.3% (ODM) and sewage sludge composts P5, P6, P7 with HA contents of 13.2%, 8.9%, 19.7% (ODM).

of these main compounds. Because of the charred grape marc a higher heat flow is measured in compost P6 compared to the other sewage sludge composts and a slight increase between 500 and 600 °C is observed (see also Fig. 4).

3.2. DSC profiles of humic acids from 10 composts

The DSC profiles of the isolated and purified humic acid fractions show an individual thermal pattern with several prominent peaks supporting the theory that humic acids are aggregates composed by different molecule moieties [21,22]. Fig. 6 illustrates DSC profiles of isolated humic acid fractions from biowaste composts and sewage sludge composts, three from each group, underlining the individual thermal behavior although similarities are obvious. Humic acids from sewage sludge composts show an additional shoulder between 300 and 400 °C. The strongest exothermic peak of isolated humic acids appears at higher temperatures (Table 3) than the second exothermic peak of the whole sample that is found between 480.5 °C (P4) and 497.1 °C (P3).

Table 3
Maxima of the two prominent exothermic peaks (°C), total carbon (TC) and total nitrogen (TN) in humic acids originating from 10 different composts; percentage of compost nitrogen bound in humic acids

HA (sample)	Peak 1 (°C)	Peak 2 (°C)	TC (%)	TN (%)	TN (% of compost N)
P1	389.0	529.2	52.5	4.77	29.3
P2	406.3	543.1	52.9	7.29	36.7
P3	426.3	540.1	51.3	8.27	44.9
P4	412.3	537.0	52.1	5.94	32.4
P5	423.8	507.0	53.9	6.07	16.7
P6	413.3	538.4	50.6	7.71	15.7
P7	419.3	517.6	54.2	5.06	17.2
P8	413.2	550.6	56.4	7.77	56.5
P9	401.2	554.5	55.0	7.31	62.3
P10	421.3	545.3	53.8	7.78	46.1

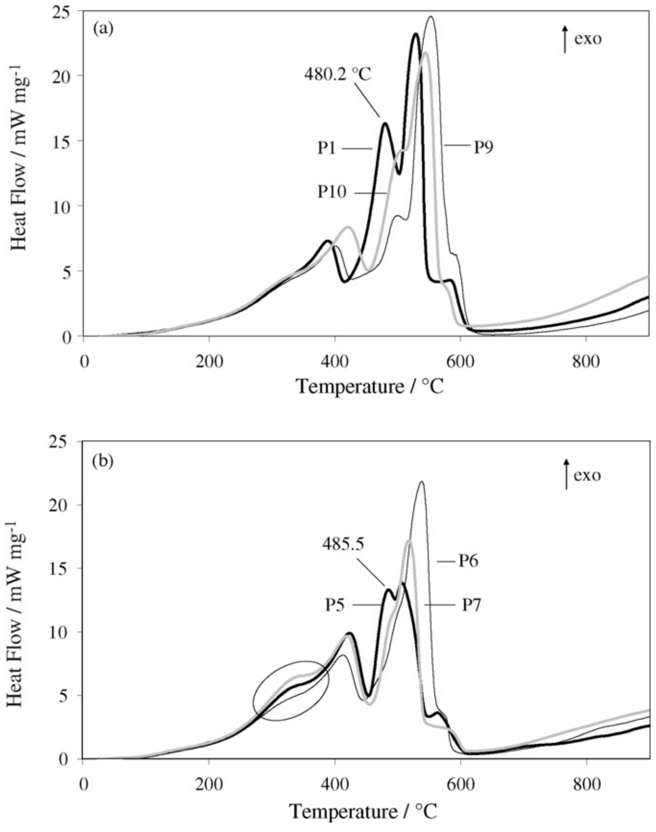

Fig. 6. DSC profiles of HA from biowaste composts P1, P9, P10 (a) and from sewage sludge composts P5, P6, P7 (b).

Table 3 compiles several data of the humic acids (HA) such as temperatures of the two prominent exothermic heat flow peaks, carbon and nitrogen contents of humic acids. The percentage of compost nitrogen bound in humic acids was calculated. There is no strong correlation ($R^2 = 0.54$, $P = 0.10$) between the nitrogen contents in the composts and those found in the humic acid fraction. It can be assumed that higher nitrogen contents in compost organic matter can cause an increase of nitrogen contents in humic acids [5]. However, systematic studies on the role of input materials with regard to nitrogen fixation in humified matter are still missing. Temperatures of additional prominent peaks in P1 and P5 are indicated in Fig. 6a and b.

Enthalpies of the compost samples, corresponding humic acids, and the residues after alkaline extraction are shown in Table 4. Enthalpies of composts and humic acids refer to dry matter (DM) and to organic dry matter ODM.

Enthalpies of the humic acid fraction are high and contribute to the energy content of the whole compost sample (Fig. 2). Therefore, enthalpies of residues after humic acids extraction are lower compared to the whole compost sample. However, enthalpies of compost organic matter are higher than enthalpies of humic acids. It is obvious that other organic compounds also contribute to these higher enthalpy values. It can primarily be assigned to soil organic matter. Due to the ingredients (yard waste, plant materials) composts always contain earth to a certain extent. Not extractable humified matter (humins) and adsorbed biomolecules in the soil matrix such as lipids and aliphatic compounds [23] can be responsible for the high values. Enthalpies of composts P4 (HA content 26.6% ODM), P5 (HA content 13.2% ODM), and P10 (31.6% ODM) show the highest enthalpy referring to organic matter. According to the specification composts P4, P5, P10 were mixed with an additional amount of earth which is confirmed by their relatively low organic matter contents.

Table 4
Enthalpies (J g^{-1}) of humic acids (HA) and composts referring to dry matter and organic dry matter (ODM), and residues after alkaline extraction referring to dry matter

Sample	HA (J g^{-1})	HA (J g^{-1}) ODM	Compost (J g^{-1})	Compost (J g^{-1}) ODM	Residues (J g^{-1})
P1	16202	17640	6584	19251	5856
P2	16247	18133	7625	20119	5835
P3	15917	17143	7370	17548	6499
P4	14996	16203	4620	23938	4172
P5	14115	15806	5063	23332	4916
P6	15313	17014	7729	18714	6987
P7	13865	15474	5312	19458	4836
P8	16951	18942	6731	21713	5320
P9	16707	19248	6288	21833	5265
P10	15832	17611	5547	26165	4730

4. Conclusions

Independent of the nature of the extracted humic acid fraction, transformation processes are indicated by a shift towards higher temperatures that can not only be explained by enrichment of not degraded substances such as wooden compounds. In contrast, due to degradation heat flow peaks of degraded wood are located at lower temperatures as shown in Fig. 4. Furthermore, degradation of biogenic waste materials during composting leads to decreasing intensities of heat flow peaks. Humification is indicated by the increase of the second exothermic peak if heat flows are referring to organic matter. Humification is not reflected by usually applied parameters such as loss of ignition or total organic carbon. For humic acid determination time-consuming analyses are necessary. Thus thermal features provide more comprehensive information on compost organic matter quality.

The influence of compost ingredients on the heat flow enables discrimination of different compost classes (sewage sludge and biowaste compost) according to their specific purpose, which is not possible by sum parameters. Measurement of heat flows and enthalpies of the transformed organic matter fraction can be a promising method to reveal the composition and to evaluate the quality of waste organic matter with regard to humification. Further investigations concentrate on multivariate data analysis and the development of classification models based on thermal data that could improve the applicability in practice. Classification models will focus on biogenic waste composition and humic acid contents.

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