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IHSS 16: FUNCTIONS OF NATURAL ORGANIC MATTER IN CHANGING ENVIRONMENT

Changes in selected hydrophobic components during composting of municipal solid wastes

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Abstract
Purpose One of the most practical ways to utilise municipal solid waste is composting, thereby producing materials that may be productively used to improve soil properties. Wastes, as well as mature composts, contain hydrophobic substances, including fats, which are more resistant to microbiological decomposition than other constituents. The aim of this work was to determine qualitative and quantitative changes of hydrophobic substances, especially fatty acids, during the course of municipal solid waste composting. This provides new information on intensity of hydrophobic versus other substances decomposition undergoing during these processes.
Materials and methods Raw materials, prepared according to MUT-DANO technology, were composted in a pile, and samples were taken after 1, 14, 28, 42, 56, 90 and 180 days of the composting. Temperature, moisture, total organic carbon, hydrophobic substances carbon (HSC) and fatty acid carbon (FAC) contents were determined in all samples. Hydrophobic substances were extracted with 1:2 (*v*/*v*) mixture of ethanol/benzene, while fats were extracted with petroleum ether and determined by GC analysis after transesterification with BF_3 in methanol.
Results and discussion The HSC decreased from 27.8 to 9.3 g kg^{-1} during first 90 days of composting, and thereafter remained constant. Similarly, the highest content of FAC was in raw compost, while the lowest was after 90 days. Octadecenoic acid predominated in the raw compost and decreased from 56 to 23 % FAC after 180 days. During the composting processes, domination of octadecenoic acid was replaced by hexadecanoic and octadecanoic acids, which increased from 18.8 to 36.7 % and 8.3 to 19.4 % FAC, respectively. The share of hexadecanoic, eicosanoic and docosanoic acids increased after the thermophilic phase. The presence of odd-numbered fatty acids (pentadecanoic and heptadecanoic) was noted, which are known to be products of the bacterial transformation-synthesis of lipid substances.
Conclusions The extent of decomposition of hydrophobic substances, especially fatty acids, is greater than other components in composted municipal solid waste, and intensity of the biotransformation is significantly correlated with composting parameters, mainly temperature and time. During the thermophilic phase of municipal solid waste composting, the decrease in total content of hydrophobic substances is approximately fivefold, while the reduction in fatty acids can be about tenfold. Unsaturated fatty acids are more intensively decomposed during the composting processes, while saturated fatty acids are more resistant. Moreover, transformation of fatty matter may result in the creation of specific isomers with odd numbers of carbon atoms.

Keywords Compost · Fatty acids · Hydrophobic substances · Municipal solid waste

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1 Introduction

One of the most practical ways to utilise municipal solid waste is composting, thereby producing materials that may be productively used to improve soil properties (Weber et al. 2007). Municipal solid waste (MSW) have variable composition, with the main ingredients being: comestible waste products (40–60 %); paper and cardboard (20 %); and glass, plastic and metal up to 40 % (Costa et al. 1991). During composting, some of these materials are biotransformed into more stable products that are rich in humic substances. According to numerous studies, the quality and quantity of these humic materials influences the stability and maturity of the final product (Garcia et al. 1992; Spaccini and Piccolo 2009). However, the actual structure of the humic substances remains controversial (de Leeuw and Largeau 1993). Most studies suggest a predominance of aromatic units in matured humic substances, while other results indicate largely aliphatic structures in humic extracts. These dissimilarities may result from differing microbial activities, especially in resynthesis processes occurring during composting (Gea et al. 2007).

Composted MSW may contain variable amounts of hydrophobic constituents, such as lipids, fats, waxes, resins, which in soil science are determined as bitumen fraction of humin. Many authors indicate these substances as an integral part of the organic matter present in municipal wastes (Amir et al. 2006; Gea et al. 2007; Réveillé et al. 2003; Veeken et al. 2000). Fats and oils originating from common households consist of straight-chain fatty acids in the form of glycerol esters (Ruggieri et al. 2008; Steger et al. 2003; Wakelin and Forster 1997). Fatty acids and lipids are integral constituents of cellular membranes, leftovers and cellular secretions, and participate in many biological processes (Klamer and Bååth 2004; Ryckeboer et al. 2003). The main sources of these substances in municipal wastes are food refuse and kitchen waste, which contain varying amounts of animal and plant fats (Komilis and Ham 2003; Maliki and Lai 2011). Due to difficulties in monitoring the transformation of lipids and fatty acids into humic substances, some authors consider the role of these substances in humification processes as mainly that of energy donors for microorganisms (Chen et al. 1997; Komilis and Ham 2003; Ryckeboer et al. 2003). Conversely, the results of other studies indicate that fatty acids play an integral part in the formation of humic substances and are important during the creation of humic acid aliphatic structures (Amir et al. 2006; Hachicha et al. 2009; Lguirati et al. 2005; Spaccini and Piccolo 2009). From this point of view, qualitative and quantitative changes in fatty acids during composting may be considered as chemical indices of compost maturity (Barje et al. 2008; Gea et al. 2007; Hachicha et al. 2009; Ruggieri et al. 2008).

As a result of its chemical properties, complicated structure and hydrophobic character, the biotransformation and decomposition of fats are dependent on several factors. The most important of these are microbiological activity, temperature, pH, humidity and the nature of the macro- and microelements (Amir et al. 2008; Barje et al. 2008; Gea et al. 2007). Many studies have reported the biotransformation of fats, although the intensity of their biodegradation is generally limited by unfavourable physiochemical properties, such as insolubility in water (Becker et al. 1999; Lefebvre et al. 1998). Routine composting technologies enable the biotransformation of wastes enriched with fats, providing these fats do not exceed 15 % of the dry mass (Filippi et al. 2002; Garcia-Gomez et al. 2003). However, this may cause an extended thermophilic phase, as a result of the high chemical energy of the fatty acids (Nakano and Matsumura 2001). Lipid biotransformation and degradation processes are usually most intensive in the thermophilic phase, during which time decreases of 80–90 % from initial values have been observed, with reductions of up to 97 % occurring during long composting periods (Baddi et al. 2004; Ruggieri et al. 2008). Biological treatment of fats under thermophilic conditions is considered to be the appropriate method for producing the required changes in the physical and chemical properties of these hydrophobic compounds (Hachicha et al. 2009; Gea et al. 2007). From this perspective, composting can be considered as an alternative method for the effective treatment of fats and oils.

Wastes, as well as mature compost, contain hydrophobic substances, including fats, which biodegradability is considered as largely limited due to unfavourable properties, such as insolubility in water (Gea et al. 2007; Lefebvre et al. 1998; Ruggieri et al. 2008). However, their biotransformation during composting processes may be influenced by other factors, mainly temperature, oxygenation and mineral composition (Barje et al. 2008). The aim of the present study was to determine qualitative and quantitative changes of hydrophobic substances, especially fatty acids, during the course of MSW composting. This provides new information on intensity of hydrophobic versus other substances decomposition undergoing during these processes.

2 Materials and methods

2.1 Compost samples

The presented research concerns material processed at the Katowice commercial composting plant (Upper Silesia, Poland), where randomly collected raw material was composted for 180 days in a pile, after initial biostabilisation according to MUT-DANO technology. Changes in temperature were measured each day throughout the composting process. Samples for analysis were taken after days 1, 14, 28, 42, 56, 90 and 180 of the composting from three different places of the pile and then averaged. The weight of each averaged samples was approximately 2.5–3.0 kg of fresh mass. All anthropogenic contaminations (glass, plastic, metal,

Table 1 Correlation coefficients between TOC, HSC, FAC and composting parameters

Parameter	TOC	HSC	FAC
Composting time	−0.97*	−0.84*	−0.70*
Temperature	−0.87*	−0.94*	−0.83*

*$p<0.05$, correlation significance

etc.) were removed manually. The moisture was determined in collected material while for further determinations, samples were dried, grounded and sieved through a sieve with diameter 2.0 mm.

2.2 Total organic carbon, hydrophobic substances carbon and fatty acid carbon

Total organic carbon (TOC) content was determined with automatic analyzer (Ströhlein CS-MAT 5500, Germany). Hydrophobic substances were extracted by means of an ethanol and benzene mixture (1:2 *v*/*v*) and fats were extracted with petroleum ether, using the Soxhlet extractor. After extraction, samples were dried in controlled condition at temperature 40 °C for 24 h to evaporate extractant. Hydrophobic substances carbon (HSC) was calculated as the difference in organic carbon before and after extraction and fatty acid carbon (FAC), determined with automatic analyzer (Ströhlein CS-MAT 5500, Germany).

2.3 GC analysis

To analyse qualitative and quantitative changes of fatty acids during compost maturation, catalysed transesterification with BF_3 in methanol method was used (Metcalfe and Schmitz 1961). In short, fats from petroleum ether extract were hydrolysed with 1 M NaOH in methanol and then esterified after addition of BF_3 14 % solution in methanol. Obtained fatty acids methyl esters (FAMEs) were isolated with hexane and identified using an gas chromatograph (GC)/mass spectrometer (MS) apparatus (Agilent 6890N gas chromatograph coupled with 5973 MS detector), according to their mass spectra. After qualitative identification, the relative FAMEs content in each sample was assessed in GC using an Agilent 6890N gas chromatograph equipped with the flame ionization detector (FID). In the FID mode, the presence of the main FAMEs isomers was confirmed with the authentic samples of hexadecanoic (i.e. palmitic), octadecanoic (i.e. stearic), oleic, linoleic and linolenic methyl esters. Thus, the remaining minor FAMEs peaks with identical molecular ions were ascribed to the isomeric methyl esters, most likely belonging to *iso* and *anteiso* series typical for bacteria.

2.4 Statistical analysis

TOC, HSC and FAC were determined by three repetitions for each sample. Mean values, presented in tables or on figures, were used to characterise the transformation processes. Statistical analyses presented by correlation coefficients between TOC, HSC, FAC and composting parameters were calculated on a basis of all obtained results using the 'Statistica 7' package at the $p<0.05$ level (Table 1).

3 Results and discussion

3.1 TOC, HSC and FAC transformation

The intensity of organic matter biotransformation and decomposition processes during composting is controlled by temperature, humidity, pH and the properties of the organic and mineral substances (Licznar et al. 2010), which affect microbiological activity in the composted material. Over the 180 days of the experiment, the amount of TOC decreased from 201.7 to 100.1 g kg^{-1} (Table 2), with the greatest intensity of this process being observed during the thermophilic composting phase (Fig. 1). This phase with average temperature >55 °C started on 20th day, the highest temperature (64 °C) was observed on 40th day of composting (see Fig. 1) and was prolonged until about the 60th day of the composting.

Table 2 Contents of total organic carbon (TOC), hydrophobic substances carbon (HSC) and fatty acids carbon (FAC) during the experiment

Composting time Days	TOC g kg^{-1}	HSC	FAC	Unsaturated % FAC	Saturated
1	201.7	27.8	1.98	61.9	31.5
14	192.0	21.2	1.32	54.9	37.6
28	184.9	17.7	0.76	55.0	39.0
42	165.0	13.5	0.41	46.0	46.3
56	157.4	9.3	0.34	45.7	53.8
90	132.8	5.8	0.13	39.2	56.8
180	100.1	4.5	0.17	28.7	64.8

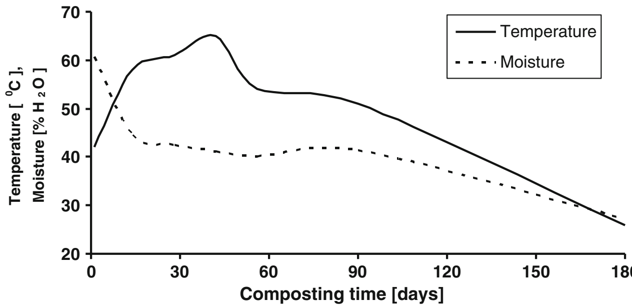

Fig. 1 Changes in temperature and humidity during composting of municipal solids wastes

The reason of a such long thermophilic phase was not sufficiently regular conversion of the material in the pile.

The HSC content decreased from 27.8 to 5.8 g kg^{-1} during the first 90 days of composting (see Table 2). After that time, it remained at the same level, due to the cooling and stabilisation of the composting material in the mesophilic phase (Chefetz et al. 1996).

More intensive changes were observed in FAC, which decreased from 1.98 to 0.13 g kg^{-1} during the first 90 days (see Table 2). After that period, the fatty acid content increased slightly, presumably as a result of stabilisation and changes in microbial composition, as suggested by Amir et al. (2008) and Ryckeboer et al. (2003). Obtained results indicate that hydrophobic substances, especially fatty acids, are transformed more intensively than other compounds of composted MSW material.

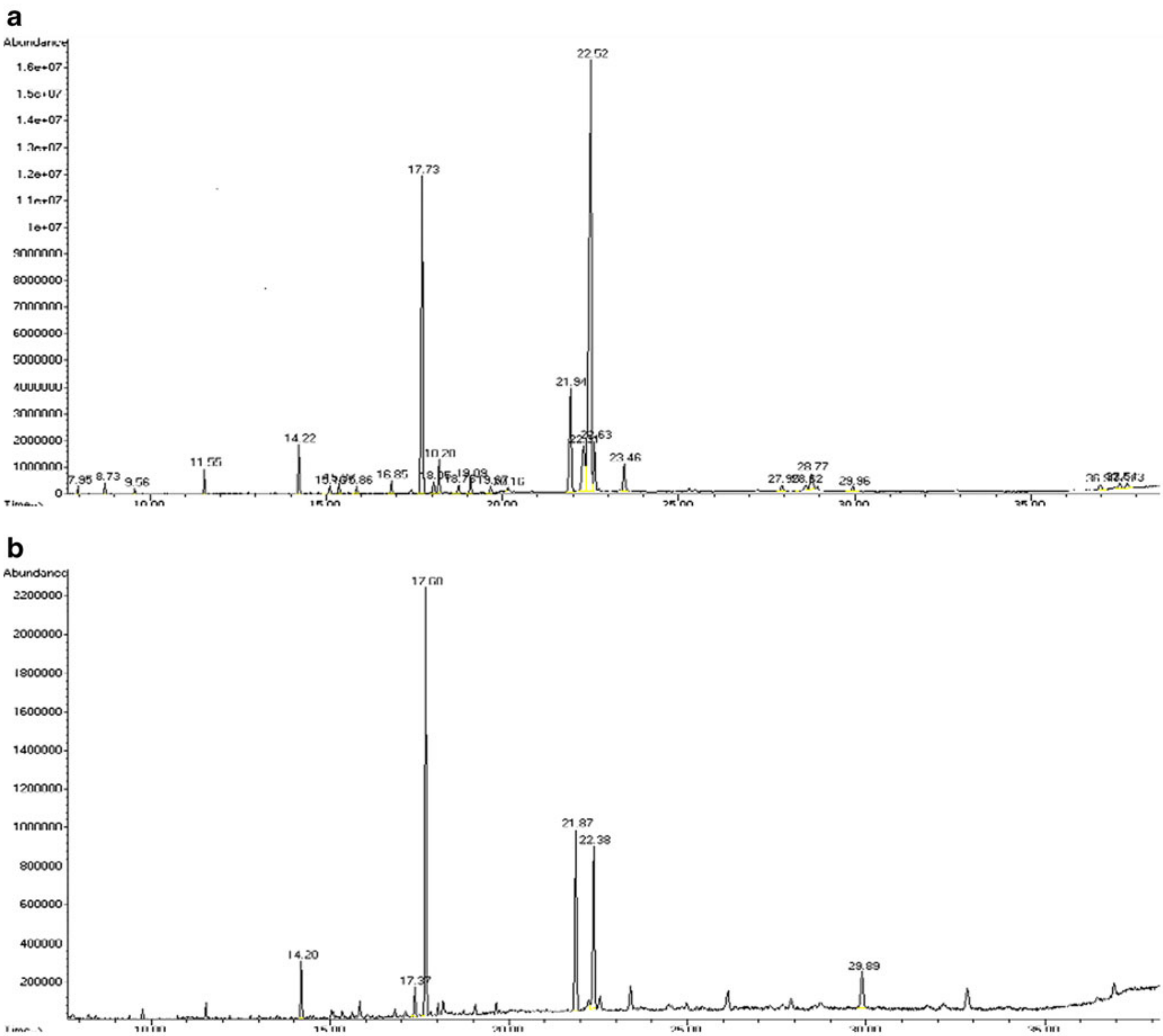

Fig. 2 Fatty acids in the capillary GC of extracted lipids at **a** the beginning of the experiment and **b** after 180 days of composting

3.2 Fatty acid transformation

During the course of the whole experiment, 24 fatty acids were identified, of which 14 were present only in trace amounts (Fig. 2). Consequently, only 10 fatty acids (six saturated and four unsaturated) were selected for detailed investigation (Tables 3 and 4).

Unsaturated fatty acids dominated in the raw material (see Table 2), which is typical for households. Octadecenoic acid (18:1) is the main lipidic component of almost all cellular membranes occurring in living organisms, and it was the main unsaturated fatty acid. Its share reached 56 % of the FAC in the raw material, and decreased to 23 % after 180 days of composting processes (see Table 3). The biggest changes were observed during the first 40 days, which correspond to the declining number of microorganisms during compost maturation (Hachicha et al. 2009; Klamer and Bååth 2004).

Despite the fact that composting processes generally lead to a decrease in FAC (see Table 2), individual fatty acids indicated varying transformation intensities. Especially during the first few weeks of composting, hexadecanoic (16:0) and octadecanoic (18:0) acids increased from 18.8 to 36.7 % and from 8.3 to 19.4 % of the total FAC, respectively (see Table 4). These acids, known as long-chain fatty acids (LCFAs), are integral components of cell membranes and usually occur in large amounts in living organisms and composted materials (Gea et al. 2007; Klamer and Bååth 2004; Ruggieri et al. 2008). The obtained results need further investigation in order to understand the release mechanisms of the various LCFAs from their parent glycerolipids, and their transformations during composting.

The role of abiotic processes in the transformation of lipid substances present in the raw material also merits further enquiry. In the presence of oxygen, unsaturated fatty acids in composted material undergo autoxidation, producing a series of hydroperoxides. These subsequently stimulate decomposition, thus contributing to the release of volatile odorous compounds, including hydrocarbons, saturated and unsaturated aldehydes, alcohols and some other oxo compounds (Frankel 1980). Similar volatile patterns were observed in the microbial decomposition of fatty foods (Ercolini et al. 2009). It is assumed that at elevated temperatures, and especially in the presence of metal ions (which act as catalysts), the autoxidation reactions must have been more intense, although the occurrence of these odorous substances was not the subject of this study. The presence of lignin-modifying basidiomycetous fungi may be considered as an additional factor influencing the formation of peroxides (Kapich et al. 2011).

Table 3 Changes in the relative amounts of unsaturated fatty acids (percent of total fatty acids) during composting

Composting time	Number of carbon atoms/number of C=C bonds			
Days	16:1	18:1	18:2	20:1
1	1.0	56.1	2.3	2.5
14	1.1	49.8	2.6	1.4
28	1.1	49.9	2.8	1.2
42	1.2	40.8	2.5	1.5
56	1.4	40.8	2.1	1.4
90	1.7	32.5	3.6	1.4
180	2.1	23.0	2.7	0.9

Table 4 Changes in the relative amounts of saturated fatty acids (percent of total fatty acids) during composting

Composting time	Number of carbon atoms/number of C=C bonds					
Days	15:0	16:0	17:0	18:0	20:0	22:0
1	1.2	18.8	1.9	8.3	0.8	0.5
14	1.5	22.3	2.1	10.3	0.9	0.8
28	1.2	24.0	1.7	10.5	0.8	0.8
42	1.5	26.5	2.2	14.0	1.1	1.0
56	2.4	30.2	2.2	15.7	1.5	1.8
90	2.3	30.2	2.3	18.9	1.3	1.8
180	2.7	36.7	2.3	19.4	1.5	2.2

In any case, both biotic and abiotic processes justify the decrease in fatty acids during the composting process. It is worth noting that, according to the results of other researchers, fungi isolated from compost do not produce these volatiles in the absence of lipid substances (Fischer et al. 1999). The observed changes and tendencies are consistent with the results of Amir et al. (2008), Gea et al. (2007) and Ruggieri et al. (2008). In the final stage of the composting processes, the fatty acids were dominated by saturated forms, primarily hexadecanoic (16:0) acid. The share of this component increased from 18.8 % at the beginning of the experiment to 36.7 % of FAC after 180 days (see Table 4).

Within the composted material some particular fatty acids with odd numbers of carbon atoms, specifically pentadecanoic (15:0) and heptadecanoic (17:0), were present, which is a characteristic of many bacteria (see Table 4). These compounds can be synthesised by bacteria de novo, or may be products of lipid matter transformation, mainly during the thermophilic phase (Řezanka and Sigler 2009). This confirms that high microbiological activity is caused by thermobasophilic bacteria (Amir et al. 2008; Ryckeboer et al. 2003), which are therefore responsible for most processes associated with the biotransformation of organic matter, including lipid substances, and especially fatty acids.

Figure 2 presents capillary gas chromatograms of FAMEs obtained from lipids extracted during various phases of the composting process. Those peaks with retention times equal to approximately 17 min correspond to saturated acids, while those of 21–22 min correspond to unsaturated fatty acids. This confirms that during the composting and maturation processes, the ratio of saturated vs. unsaturated fatty acids increases in comparison with the fresh compost.

Since the total amount of fatty acids clearly decreased during composting, the increased proportion of saturated fatty acids represents their greater resistance to decomposition than unsaturated forms. The amount of FAC slightly increased at the end of the composting, in comparison to that present after 90 days (see Table 2). Intensity of the transformation processes decrease during maturation stage, due to lowering of microbiological activity, what is typical for the stabilisation phase of compost production (Chefetz et al. 1996; Ryckeboer et al. 2003), indicating that fungi, actinomycetes, mesophilic bacteria and other microorganisms—that dominate the microbial community—are unable to transform the fatty acids.

4 Conclusions

The extent of decomposition of hydrophobic substances, especially fatty acids, is greater than other components of composted municipal solid waste, and intensity of the biotransformation is significantly correlated with composting parameters, mainly temperature and time. During the thermophilic phase of municipal solid waste composting, the decrease in total content of hydrophobic substances is approximately fivefold, while the reduction in fatty acids can be about tenfold. Unsaturated fatty acids are more intensively decomposed during the composting processes, while saturated fatty acids are more resistant. Moreover, transformation of fatty matter may result in the creation of specific isomers with odd numbers of carbon atoms.

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