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Evolution of temperature and chemical parameters during composting of the pig slurry solid fraction amended with natural zeolite

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Abstract

A 3-month experiment was conducted at a 300 kg scale to observe decomposition processes in pig slurry solids amended with two different doses of natural Slovak zeolite–clinoptilolite (substrates S1 and S2, 1% and 2% of zeolite by weight, respectively) in comparison with the control (unamended solids). The experimental and control substrates were stored outdoors in sheltered static piles at ambient temperatures ranging from 8.0 to 34.7 °C. The solid fraction (SF) of pig slurry was obtained by separation on vibration sieves prior to slurry treatment with activated sludge. The initial water content of the SF was 77.1% and no water was added to the piles during the storage.

The temperature in the core of the piles was recorded throughout the experiment. By day 3 and 5 of storage (1% and 2% zeolite, resp.), the temperature in the substrates S1 and S2 exceeded 55 °C and remained above this level for 15 days while the highest temperature recorded in the control during the experiment was 29.8 °C.

Samples from the core of the piles were taken periodically to determine pH, dry matter at 105 °C (DM), ash (550° C/4 h), ammonia nitrogen (N-NH_4^+), nitrate nitrogen (N-NO_3^-), total nitrogen (N_t), total phosphorus (P_t); total organic carbon (TOC) was computed.

The results showed that pH levels in S1 and S2 remained below that in the control for most of the thermophilic stage. This may be related to water-soluble ammonia and the affinity of zeolites to ammonium ions. A significant decrease in the level of ammonia nitrogen in water extracts from S1 and S2 was observed between days 5 and 35 in comparison with the control. The values of ash also differed and corresponded to the intensity of the decomposition processes in the respective substrates.
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1. Introduction

Processing strategies for livestock slurries produced by intensive farms with a litterless system focus on the prevention of dissemination of diseases, minimisation of environmental pollution and maximum utilisation of nutrients.

Pig farms generate large volumes of liquid wastes with a high concentration of organic matter, nutrients, trace elements, and a variety of infective agents (Ondrašovič et al., 1990; Strauch and Ballarini, 1994). These wastes have a high fertiliser value and they should therefore be utilised in plant production. They, however, are often rejected after slurry treatment (Ondrašovičová, 1998) due to agricultural difficulties. In the first stage of this treatment, pig slurry solids are separated on vibrating gravity screens, belt presses, by centrifugation, etc. Piggery solid wastes are nutrient rich fibrous materials readily amenable to aerobic composting (Bhamidimarry and Pandey, 1996). However, immediately after separation they contain a large proportion of organic substances unaccessible to plants but also high numbers

of microorganisms and parasitic stages that may survive for different periods of time. Because of that they should be subjected to biothermic treatment prior to application to cropland (Juriš et al., 1991). However, in practice the solid fraction (SF) is frequently stored in middens and yards where it matures under anaerobic conditions.

Zeolites are minerals with a unique structure which allows them to entrap or release various cations as a consequence of cation-exchange reactions and adsorption. The application of zeolites in animal production has been studied extensively from many aspects, particularly in relation to improvement of the environment in animal houses (Mumpton and Fishman, 1977; Amon et al., 1997), minimization of environmental pollution during animal waste management (Witter and Lopez-Real, 1988; Bernal et al., 1993; Kithome et al., 1998) elimination of aflatoxins (Skalická et al., 2000), and others. The role of zeolites in the anaerobic digestion of piggery wastes was investigated by Borja et al. (1993) and Milán et al. (2001). Application of zeolites to soil can improve some physical and chemical properties of the soil system, such as pH, cation exchange capacity, water holding capacity and the use of nutrients (Králová et al., 1994).

The present study was concerned with the effect of two different doses of natural zeolites on stabilization processes in pig slurry solids with regard to the development of temperature in the core of stored SF substrates and changes in selected chemical parameters from viewpoint hygiene safety of the final substrate and environmental pollution.

2. Methods

2.1. Natural zeolite

Clinoptilolite, a natural zeolite mined at Nižný Hrabovec, Slovakia, was ground to powder (fractions: 0.38%, <0.063 mm; 3.23%, 0.063–0.09 mm; 7.15%, 0.09–0.125 mm; 76.92%, 0.125–0.250 mm; 10.77%, 0.25–0.5 mm; 1.46%, 0.25–0.50 mm; 0.03%, 0.5–1.0 mm) and used in the study. It was pre-dried at 105 °C for 4 h. Some characteristics of the natural zeolite are listed in Table 1.

2.2. Substrates

The SF of pig slurry was obtained in the first stage of slurry treatment at an intensive pig farm by separation on vibration sieves. The solids were collected in a container which was transported immediately to an experimental area located at a distance of about 15 km from the farm. The initial water content of the SF was 77.1%. Four piles were set-up, two unamended controls and two zeolite-amended substrates S1 and S2 (1% and 2% zeolite by weight, respectively). Only one trial was conducted.

2.3. Construction of static piles

The experiment was conducted outdoors from April till July. The control and zeolite-amended SF was homogenized by mechanical mixing and piled up to individual compartments of a dimension of 185 cm × 93 cm. The compartments were covered with a roof about 60 cm above the top of the substrates to protect them from rain but allow sufficient access of air. The floor of the compartments consisted of concrete covered with a 15 cm layer of crushed stone on which a plastic mesh and a layer of wood chips (spruce) of 5 cm thickness was placed and the SF was piled on the chips. Air ducts allowed free access of air to the bottom of the piles but no air was forced through the latter to simulate a situation that could arise under field conditions. The total weight of each pile was 300 kg and the initial height 60–65 cm. The substrates were stored in this structure for 3 months without turning or adding water to the piles. Two temperature probes were inserted at two symmetrical locations in each pile and the ambient temperature was recorded, too.

Table 1
Some characteristics of the natural zeolite used

Chemical composition (%)		Phase composition (%)	
SiO_2	70.08	Clinoptilolite	40–50
Al_2O_3	12.29	X-ray amorphous phase	22.5–40
Fe_2O_3	0.89	Plagioclase	7.8–10.7
FeO	0.45	Cristobalite	6.3–8.7
TiO_2	0.154	Quartz	2.3–3.2
CaO	3.88	Biotite	~1
MgO	0.73	Additional accompanying	Glauconite, montmorillonite,
MnO	0.027	minerals	zircon, Apatite
P_2O_5	0.03		
Na_2O	0.48		
K_2O	3.27		
S	0.03		

2.4. Analytical determinations

The temperature in the core of the substrates was recorded by means of a programmable Commeter System (Rožnov pod Radhoštěm, Czech Republic) with appropriate probes. Samples for chemical examination were taken immediately before adding zeolite and then from the core of the substrates, approximately 10 cm to the centre from the temperature probes, after 1, 2, 5, 7, 12, 21, 28, 40, 49, 61 and 84 days of storage. The samples were examined for pH (1:10 water extract) using a pH electrode, dry matter (105 °C to a constant weight), residuum-on-ignition (550 °C/4 h), water soluble ammonium nitrogen N-NH_4^+ by titration or photometrically after steam distillation and nitrate nitrogen N-NO_3^- by an ion selective electrode and the ORION Research ion analyser Model EA 940 in the same water extract as that used for pH determination. Total organic carbon (TOC) was calculated on the basis of the loss-on-ignition according to Navarro et al. (1993). Portion of samples for N_t and P_t determinations were digested using a HACH-Digesdahl apparatus. N_t was distilled with NaOH (40%) and P_t was determined using the vanadomolybdate method. Organic nitrogen (N_t) was computed as N_t-(N-NH_4^+ + N-NO_3^-). The analyses were performed in duplicate.

Correlation coefficients and P values were computed using GraphPad Prism software to show the relationship between the control and the treated groups against decomposition time.

3. Results and discussion

3.1. Temperature

During the storage of animal manure some of the organic substances undergo decomposition and the product obtained is less odorous and also safer from the hygienic point of view. In dependence on the conditions of storage (temperature, aeration, humidity, pH) organic matter is decomposed at various speeds at several temperature stages at which specific microorganisms play a dominant role. Stentiford (1996) suggested that temperatures higher than 55 °C maximized sanitation, those between 45 and 55 °C maximized the biodegradation rates, and between 35 and 40 °C maximized microbial diversity in the composting process. According to Strauch and Ballarini (1994) only the thermophilic range of 55 °C is sufficient to destroy pathogens.

Throughout the experimental period, the ambient temperature ranged from 8 to 34.7 °C. The temperature development in the S1 and S2 piles between days 1 and 21 differed considerably from that in the control (Fig. 1). The zeolite-amended substrates showed an abrupt increase in temperature as soon as on day 2 of storage when the core temperature reached 54.4 °C in S1 and 44.1 °C in S2 at mean ambient temperature of 10.9 °C. The core temperature in S1 exceeded 55 °C on day 3 and in S2 on day 5 and persisted above this level for 15 days which sufficed to ensure devitalization of potentially present pathogens. The highest temperatures reached in S1 and S2 were 62.1 and 61.8 °C, respectively. By days 35–38, the core temperatures in S1 and S2 substrates decreased gradually to the ambient temperature and followed its course.

Although the control substrate showed an increase by about 10–18° during the first 12 days of storage as compared to the ambient temperature, the highest temperature of 29.8 °C recorded on day 3 was by no means sufficient to ensure the hygiene safety of the final material.

3.2. Chemical parameters

3.2.1. pH level

The pH level is one of the important characteristics of the composting process. Bertoldi et al. (1983) suggested that the optimum pH values for composting are between 5.5 and 8.0. During storage mineralization of organic
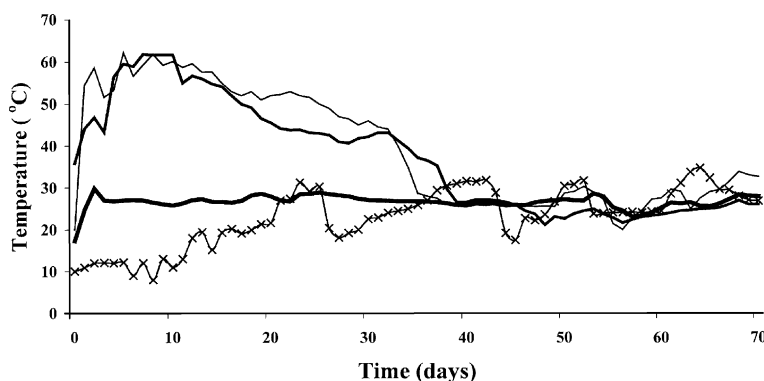

Fig. 1. Changes in ambient air temperature and temperature in the core of control and experimental piles during the composting process (■ = control piles, ─ = pile S1, 1% zeolite by weight, ─ = pile S2, 2% zeolite by weight, x = ambient temperature).
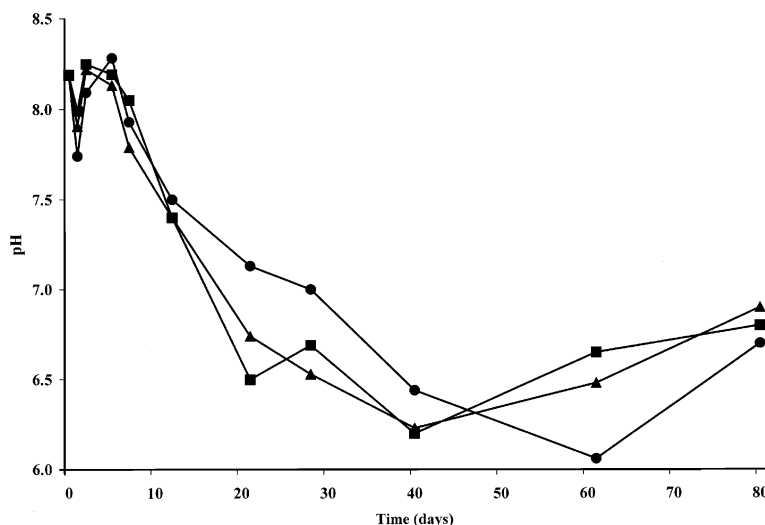
Fig. 2. Changes in pH in the core of control and experimental piles during the composting process (● = control piles, ■ = pile S1, 1% zeolite by weight, ▲ = pile S2, 2% zeolite by weight).

nitrogen to ammonia nitrogen takes place and pH values increase. A decrease in pH may be caused by increased production of organic acids or increased nitrification. The pH level affects also the ammonium exchange in zeolites as discussed later in this paper.

The pH values of all piles (Fig. 2) increased slightly by day 5 of storage and then decreased gradually up to day 40 in S1 and S2, reaching 6.2 and 6.23, respectively, and up to day 60 in the control with the lowest value of 6. After these periods the pH levels increased again to 6.8 and 6.9 in S1 and S2, respectively and to 6.7 in the control. Fig. 2 shows that the pH level in zeolite-amended substrates remained below that in the control for most of the thermophilic phase, particularly between day 20 and 40 of storage. As the concentration of water-soluble ammonia in S1 and S2 during the thermophilic stage was also below that in the control and N-NO$_3^-$ levels did not indicate intensive nitrification, the differences in pH levels between S1 and S2 and the controls suggest the influence of zeolite to be based on its affinity to ammonium ions. A high moisture content within the piles might have created anaerobic conditions in certain layers or "pockets" leading to a more acidic environment within the piles (production of fatty acids).

3.2.2. DM content

Loss of water as a result of evaporation as the process progresses is a major part of the composting process. The initial moisture content will typically lie in the range of 55–65% depending on the materials being used. The maximum amount of moisture which can be tolerated varies between starting materials being limited by the inhibition of aerobic activity. On the basis of a wide range of research results it would seem that the majority of composting processes should operate with moisture contents in the 40–60% band (Stentiford, 1996). How-

ever, there are big differences in the optimal moisture content between various materials which depend on the physical structure of the materials and their particle size (Forshell, 1993). Caceres et al. (1998) used the static composting strategy for a material with intentionally high initial moisture content (82% fresh weight) which made it possible to maintain adequate moisture throughout the process. They rapidly achieved thermophilic temperatures at an air flow of 260 $l\,min^{-1}\,m^{-3}$.

The initial water content in pig slurry solids was very high (77.1%). As no air was forced through the piles during the storage it was surprising that thermophilic temperatures could develop in this material, however, the piles were only 60 cm high. The material itself was rather fibrous and not very densely packed and some improvement in its handleability was observed after the addition of 1% and 2% zeolite. Some air could be trapped in the solids during their collection in containers and subsequent mechanical mixing and piling into the compartments but this manipulation was practically the same for all piles. Zeolite itself accounted only for 1% and 2% of additional DM which was negligible with regard to the low initial DM content. During the composting process an increase in DM is expected due to the evaporation of water and release of volatile compounds to the environment. Fig. 3 shows that within 80 days of storage the DM content increased from the initial 22.9%, 23.9% and 24.9% (control, S1 and S2) to the final 53.4%, 59.3% and 58%, respectively. Small initial increase in DM could be ascribed to the release of liquid that was retained in the piles. Only after about 30 days the DM content of 35% was reached in all piles above which no marked inhibition of microbial activity is expected (Stentiford, 1996). The high initial moisture content made it possible to maintain an adequate moisture up to the end of the experimental period. On
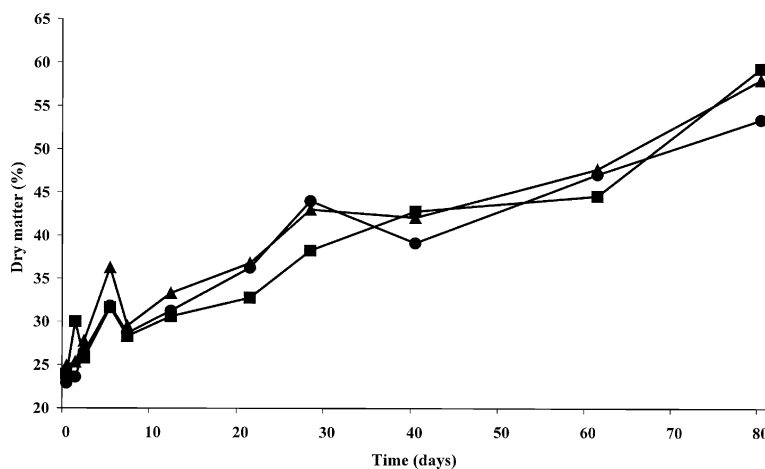
Fig. 3. Changes in DM content in the core of control and experimental piles during the composting process (● = control piles, ■ = pile S1, 1% zeolite by weight, ▲ = pile S2, 2% zeolite by weight).

the whole, fluctuations in DM may have been caused also by natural variation.

3.2.3. Ash

Ash (550 °C) is the inorganic portion of substrates principally composed of a variety of inorganic minerals such as calcium, magnesium, sodium, iron and manganese along with other trace metals. According to Day et al. (1998) these cations are usually associated with carbonates, bicarbonates, sulphates, phosphates, nitrites, etc. Because these materials are generally unaffected by biological action, they should pass through the composting process unaltered. They may, however, influence the biodegradability of the organic fraction. Because the biological breakdown of organic material involves the consumption of oxygen and the production of metabolic water and carbon dioxide, a net loss in organic matter occurs and the inorganic content of the material increases as the composting process progresses.

The results obtained are illustrated in Fig. 4. The levels of this parameter show a notable difference between the control and the S1 and S2 substrates which corresponds to the intensity of decomposition processes indicated by the temperature development.

3.2.4. Ammonium nitrogen

Nitrogen transformations in the first stage of composting of nitrogen-rich material are generally characterized by high rates of ammonification. The greatest nitrogen losses during composting were caused by gaseous emissions in the form of NH_3. High losses of ammonium nitrogen not only reduce the agronomic value of the end-product and represent a waste of a valuable resource, but also contribute to the pollution of the environment (Witter and Lopez-Real, 1988).

High affinity to ammonia ions is a well-known feature of zeolites. Zeolite highly increased ammonia adsorption when added to pig slurry solids (Vargová et al., 2002).
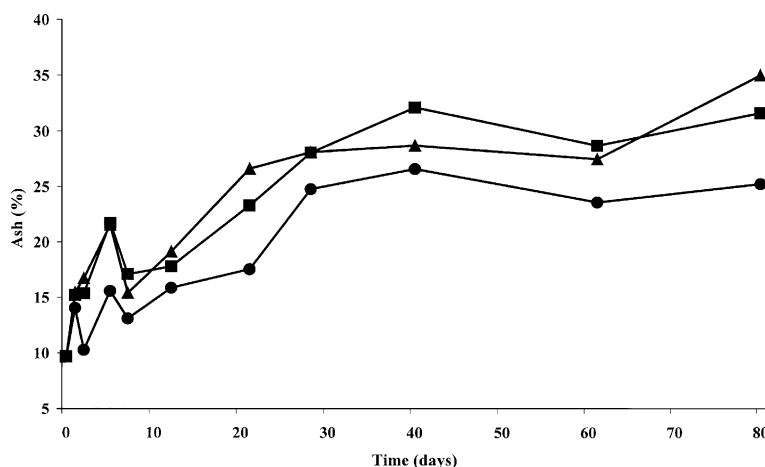

Fig. 4. Changes in ash in the core of control and experimental piles during the composting process (● = control piles, ■ = pile S1, 1% zeolite by weight, ▲ = pile S2, 2% zeolite by weight).

Bernal and Lopez-Real (1993) reported that maximum adsorption of ammonia in clinoptilolite zeolites they tested was between 6.255 and 14.155 mg N g^{-1}. Koon and Kaufmann (1975) investigated the impact of pH on the ammonium exchange in zeolites and obtained the highest ammonium exchange at pH 6. They explained that at low pH, the ammonium ions had to compete with hydrogen ions among the exchange sites, however, when the pH was high, the ammonium ions were transformed to ammonia gas. For practical applications, they recommended a pH value within the interval 4–8 during the loading phase. Witter and Kirchmann (1989) found the ammonium-adsorption capacity of zeolite clinoptilolite to be more than 10 times greater than the ammonia adsorption. Kithome et al. (1998) found that when performing experiments with pH varying between 4 and 7, most ammonium was adsorbed at pH 7. They

gave the same explanation for low pH as Koon and Kaufmann (1975) but assumed that new sorption sites were formed at a higher pH. The initial NH_4^+ concentration and ionic-strength competition were named among additional important factors with regard to ammonium exchange by zeolites (Koon and Kaufmann, 1975; Kithome et al., 1998).

Hlavay et al. (1982) investigated the impact of grain size distribution on ammonium exchange capacity in the intervals of 0.5–1.0, 0.3–1.6 and 1.6–4.0 mm. The smallest fraction resulted in the highest ammonium exchange capacity.

It has been noted (Tiquia et al., 1997) that the absence of or a decrease in $N-NH_4^+$ is an indicator of both of good composting and maturation process.

Our results showed a significant effect of zeolite on the level of ammonium nitrogen in water extracts
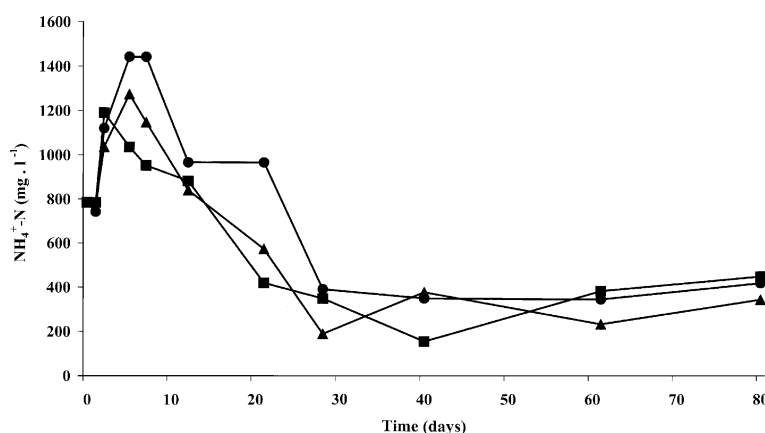

Fig. 5. Changes in ammonium nitrogen level in water extracts of control and experimental piles during the composting process ($\bullet$ = control piles, $\blacksquare$ = pile S1, 1% zeolite by weight, $\blacktriangle$ = pile S2, 2% zeolite by weight).

Table 2
Changes in chemical properties of the control and experimental substrates with time[a]

Parameter	Treatment	Day 0	Day 5	Day 12	Day 28	Day 40	Day 84
$N-NO_3^-$ mg 100 g^{-1} dw	Control	0.1	1.1	1.6	1.3	0.2	5.3
	S1		1.4	0.8	0.3	0.1	3.1
	S2		0.7	0.8	0.3	0.1	8.7
C_{org}% dw	Control	46.01	43.03	42.91	38.33	35.62	38.12
	S1		39.90	41.93	36.75	34.60	34.92
	S2		40.02	41.24	36.60	36.30	33.10
N_{org}% dw	Control	2.38	1.34	1.60	2.23	2.76	2.88
	S1		1.27	1.33	2.09	2.75	2.78
	S2		1.07	1.32	2.47	3.04	2.80
C_{org}/N_{org}	Control	19.3	32.1	26.8	17.2	12.9	13.2
	S1		31.4	31.5	17.6	12.6	12.6
	S2		37.4	31.2	14.8	11.9	11.8
P_t mg 100 g^{-1} dw	Control	42.7	66.9	90.0	163.4	201.4	404.6
	S1		60.4	71.6	143.5	225.7	505.5
	S2		65.7	79.2	169.4	239.6	426.0

[a] Means of four duplicates for the control and two duplicates for S1 and S2 are shown control = unamended SF; S1 = SF + 1% zeolite by weight; S2 = SF + 2% zeolite by weight dw = results are reported on dry weight.

Table 3
Correlation coefficient values between chemical parameters determined in pig slurry solid fraction

	pH C	pH 1% Z	pH 2% Z	DM C	DM 1% Z	DM 2% Z	Ash C	Ash 1% Z	Ash 2% Z	NH_4^+ C	NH_4^+ 1% Z	NH_4^+ 2% Z	N_t C	N_t 1% Z	N_t 2% Z	P_t C	P_t 1% Z	P_t 2% Z
pH 1% Z	0.92***																	
pH 2% Z	0.94***	0.99***																
DM C	−0.83***	−0.79**	−0.80**															
DM 1% Z	−0.79**	−0.69**	−0.69**	0.92***														
DM 2% Z	−0.79**	−0.74**	−0.75**	0.99***	0.94***													
Ash C	−0.89***	−0.89***	−0.91***	0.90***	0.86***	0.89***												
Ash 1% Z	−0.85***	−0.85***	−0.87***	0.92***	0.87***	0.91***	0.97***											
Ash 2% Z	−0.81**	−0.84***	−0.83***	0.94***	0.89***	0.95***	0.92***	0.96***										
NH_4^+ C	0.82**	0.73**	0.74**	−0.64*	−0.68*	−0.61*	−0.75**	−0.62*	−0.59*									
NH_4^+ 1% Z	0.88***	0.93***	0.93***	−0.71**	−0.68*	−0.67*	−0.86***	−0.78**	−0.73**	0.83***								
NH_4^+ 2% Z	0.88***	0.86***	0.87***	−0.76**	−0.70**	−0.70**	−0.82**	−0.71**	−0.70**	0.95***	0.91***							
N_t C	−0.91***	−0.89***	−0.88***	0.87***	0.74**	0.83***	0.83***	0.84***	0.84***	−0.70**	−0.84***	−0.84***						
N_t 1% Z	−0.82**	−0.84***	−0.82***	0.82***	0.85***	0.84***	0.88***	0.88***	0.85***	−0.73**	−0.85***	−0.77**	0.80**					
N_t 2% Z	−0.86***	−0.73**	−0.80**	0.80**	0.74**	0.76**	0.88***	0.80**	0.71**	−0.85***	−0.79**	−0.86***	0.80**	0.74**				
P_t C	−0.90***	−0.76**	−0.77**	0.92***	0.89***	0.91***	0.82**	0.83***	0.83***	−0.70**	−0.71**	−0.76**	0,90***	0.82**	0.81***			
P_t 1% Z	−0.76**	−0.67*	−0.65*	0.81**	0.91***	0.85***	0.75**	0.79**	0.79**	−0.60*	−0.64*	−0.61*	0.72**	0.90***	0.62*	0.89***		
P_t 2% Z	−0.92***	−0.79**	−0.80**	0.92***	0.92***	0.91***	0.87***	0.86***	0.84***	−0.76**	−0.77**	−0.80**	0.89***	0.88***	0.85***	0.99***	0.92***	

Correlations were based on 12 average data of all piles during the 84-day study: ***, ** and * indicate correlation significant at 0.001, 0.01 and 0.05 probability levels, respectively.

(Fig. 5). During the thermophilic stage ammonia nitrogen concentration in water extracts from the control piles exceeded considerably that in the S1 and S2 piles. The greatest decrease in comparison with the control was observed on days 5, 7 and 21 (by 406, 490 and 546 mg l^{-1}, respectively) in S1 and on days 7, 21 and 28 (by 294, 392 and 329 mg l^{-1} respectively) in S2. The pH levels during most of the storage were between 6 and 7.5, the range supporting maximum ion exchange. Adsorption and subsequent slow release of ammonia to the microenvironment of bacteria in the experimental S1 and S2 piles may be one of the factors that can affect the decomposition process in the piles and, at the same time, decrease the loss of nutrients from pig slurry solids stored in field heaps.

3.2.5. Nitrate nitrogen, organic C and N, and total P

Additional results of chemical analysis are presented in Table 2. There was no nitrification during the thermophilic stage since (1) the nitrification bacteria are limited by temperatures ≈45 °C and (2) at high temperature competition for oxygen (for aerobic respiration) was so hard that nitrification was not possible. This is in agreement with Edwards and Daniel (1992).

The C/N ratio is one of the vital aspects of composting. Values between 25:1 and 35:1 are considered to be optimal. If the C/N ratio exceeds 35:1 the composting process is slowed down, while a ratio less than 20:1 results in a loss of nitrogen which occurs mainly as NH$_3$ gaseous emissions (Martins and Dewes, 1992). The bioavailabilty of especially carbon is also important. During the composting process the biologically degradable organic matter is converted into volatile CO$_2$ and H$_2$O and is removed from the compost and the total N content increases which results in a C/N decrease toward the end of composting (Vuorinen and Saharinen, 1997; Day et al., 1998). According to Paré et al. (1998) C/N ratios should be based on biologically available N and C and not on total N and C. The Table 2 indicates that the initial C$_{org}$/N$_{org}$ determined in the SF was 19.3 which is not unusual for animal manure. To supply additional C and decrease excessive moisture addition the of bulking agents is recommended, such as straw or sawdust. Although no bulking agent was added, the C$_{org}$/N$_{org}$ increased within 5 days to the optimum range and then decreased gradually to values ranging from 11.8 to 13.2 which is common for mature composts. As the results are reported on a dry weight basis, the loss of moisture also affected the results.

In this context we should mention the observation of Keyser et al. (1978) that addition of N sources containing N-NH$_4^+$ to materials with a limited content of easily biodegradable C can exert negative effects on microbial activity by disturbing the outcome of the competition between potent and less potent decomposers through "ammonia metabolite repression". In a material such as

the SF examined, adsorption of ammonia nitrogen by zeolites in the initial stage of composting may have eliminated to a certain degree such a repression and increased the activity of thermophilic microorganisms. Clay minerals and other surface-active particles have been reported to influence microbial and enzymatic transformation of a variety of substances, including ammonium, sulphur, carbohydrates, proteinaceous materials and phenolic compounds. Zeolite counteracted to some extent the inhibitory effect of ammonia during thermophilic anaerobic digestion of cattle manure and had a stimulatory effect leading to a decreased lag phase (Borja et al., 1993).

The initial P$_t$ content was low (42.7 mg 100 g^{-1} dw). It increased about 10 times by the end of the experiment due mainly to the loss of carbon as carbon dioxide emissions (Vuorinen and Saharinen, 1997).

The computed correlation coefficients and the respective P values summarized in Table 3 show a significant correlation between most chemical parameters determined as well as between the control and the S1 and S2 substrates.

In general there were no significant differences in the effect of 1% and 2% zeolite doses. With regard to economy aspects we could recommend the lower dose.

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