Investigating Natural Zeolite and Wood Ash Effects on Carbon and Nitrogen Content in Grain Residue Compost

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Abstract

The key aim of this work is to analyze and evaluate the possibilities of using fermented wastes (grain residue) for composting, taking into account carbon and nitrogen. The key performance task was to carry out research on composting fermented waste by inserting natural zeolite or biofuel ash and using a dynamic method in designed equipment and to determine the quality of the obtained compost with regard to its suitability for fertilization. The investigations were performed using laboratory composting equipment (Lithuanian patent No. 2008 081). The compost's composition (the content of nitrogen and carbon), humidity, and pH were determined weekly.

Upon using an additive of natural zeolite, losses of nitrogen decreased 65.6% and 84.0% – when zeolite and biofuel ash mixture was used. During the investigations differences between carbon content in the control specimens and in the specimens of compost with zeolite and with ash were identified and, on average, reached 51%, and in the specimens containing only zeolite – 20%. As the investigations of grain composting show, 1 kg of grain compost mixed with apple-tree leaves produced, on average, 94.25 mg/m³ NH_4. When additives zeolite and zeolite with ash were used, this amount decreased up to 1.2 and 2.11 times, respectively. The same tendencies were also established for ammonia release from compost. When grain was composted together with leaves, 1 kg of compost released 58.5 mg/m³ CH_4, H_2S – 25.68 mg/m³ and 15.67 mg/m³ of VOC during 4 weeks. It is determined that the use of natural zeolite reduced CH_4 emissions 1.16 times and H_2S emissions 1.28 times, and upon using zeolite with ash these differences reached 1.25 and 1.47 times, respectively.

Keywords: biofuel ash, carbon, composting, grain residue, nitrogen, natural zeolite

Introduction

Efforts to transition to a climate-friendly economy based on the adjustment of CO_2 emission technologies and renewable energy sources have brought to light one of the most cost-efficient methods of waste recovery: biogas production.

With bio-ethanol production volumes growing, the amount of waste from grain processing, distillers grains, will also inevitably grow. Lithuania, like other EU countries, uses Poaceae grains (wheat, triticale, barley, or, less frequently, rye) and their mixtures for bio-ethanol production. In the process of grain fermentation into alcohol, one third of dry matter contained therein is regenerated into by-products. At the end of fermentation two main products (distillers) remain: a non-fermented fraction (coarser parts of grains) and a fine liquid fraction comprising very small

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grain particles, yeast, and soluble nutrients. At the end of the production process the non-fermented fraction is most frequently mixed with the fine liquid fraction and this product is called distillers grains with soluble residues. It is a valuable source of energy, proteins, and minerals. After processing 1 t of grains into bio-ethanol, around 3-3.8 t of fresh humid distillers grains with an 8-10% dry mass (DM) content remain in the plant. In Lithuania humid distillers grains are dewatered up to 70-75% humidity (25-30% DM). In foreign countries distiller grains are humidified up to 40-50% DM, and around half of them are dried and used as a protein additive to produce compound feeds [1]. With bio-ethanol production volumes growing, the amount of distillers grains will also grow and therefore their handling will become one of the most urgent tasks.

Due to the high content of potassium and low content of phosphorus, fertilization with non-treated distillers grains is not recommended. Taking account of distillers grains' pH, varying from 3.5 to 4, and chemical composition, scientists from the Lithuanian University of Agriculture (LUA) [2] performed a complex evaluation of the impact of fertilization with distillers grains on plant productivity and quality, changes in agrochemical properties of soil and leakage of biogenic substances to the environment, and determined that the most suitable rate of fertilization with grains, in terms of cost-effectiveness and ecology, would be 50 t/ha [3]. One of the greatest concerns during fertilization is to avoid nitrogen losses, which will mainly result from spread and non-inserted fertilizers during the dry and warm season of the year. For example, nitrogen losses from liquid fertilizer (manure) vary from 3 to 50%. But upon spreading manure in late autumn, nitrogen losses from the fertilizer not inserted during 24 hours account for only 10%. In the meantime, slurry, spread in early spring but not inserted, may lose up to 10% of ammonia nitrogen in an hour, up to 20% in a day, and up to 40% if not inserted at all [4, 5].

In order to avoid nitrogen and carbon losses, waste from biogas recovery has to be additionally stabilized by composting. Composting of biodegradable waste is the treatment of different types of biodegradable waste either dynamically or statically. This treatment means a biochemical process during which compounds of organic substances, with the help of microorganisms and oxygen, are transformed into minerals [6].

The most widely applied technologies used in the most important composting units are: static pile [7], aerated static pile [8], agitated bed [9], rotary drum [10], and compost bin [11]. Various techniques, including Biotank, Frezer-Ivensen, Irp-Tomas, Festilia, Trigos, and Dano [12], are employed worldwide to treat large amounts of biodegradable wastes and produce compost. Interest in low-capacity composting equipment, which is not useful in terms of economy, has currently been growing. However, with waste transport costs growing, the pay-back time of this equipment should considerably shorten [13]. Furthermore, with advanced and low-waste technologies developing, the cost of low-capacity equipment becomes lower. In the process of composting, structural materials allowing the stabilization and control of gaseous emission processes are most fre-

quently used [14]. One of the possible additives suitable for compost stabilization is bio-fuel ash. Lithuania can annually prepare around 5 million m³ of forest fuel, which should generate around 25-30 thousand tons of ash after being burnt; however, this would create the problems of its use and treatment [15]. Several areas of forest fuel ash use are known in global practice [16]. Scandinavia (particularly Sweden) applies non-traditional ash management – ash is mixed into building and road paving mixtures [17, 18]. But since ancient times ash has been used as the source of Ca, Mg, K, P, and micro-elements for agricultural crops [19, 20].

Another structural material that is widely applied in many areas, including environmental protection, is zeolite – a natural mineral. One of the most widely used types of zeolite is clinoptilolite. After modifying zeolite (clinoptilolite) by a special technique, a number of certified products of agricultural purpose are produced. One of them is *ZeoVit* sorbent used in waste water and slurry treatment technologies, sludge management, and bio-technologies, and collection of heavy metals, gas, radioactive pollutants, and other harmful emissions [21]. As confirmed by investigations carried out in Lithuania, the sorbent allows efficient dealing with environmental problems related to the reduction of nitrogen pollution from agricultural facilities (livestock and poultry farms); manure, slurry and waste water management; suppression of unpleasant odours and undesirable gas emissions from sewage sludge, landfills and other sites; and reducing the concentration of mobile forms of heavy metals in sludge, waste water, and contaminated soil [14]. Scientists from the Lithuanian Institute of Horticulture [22] proved that the zeolite soil improvement product *ZeoVit* efficiently increased the productivity of vegetables. Among the first scientists who noticed the benefit of zeolite in agriculture were M. A. Weber [23], F. A. Mumpton [24], and D. B. Hawkins [25]. Other foreign scientists [26-28] investigated the possibilities that allow reductions in the content of pollutants (heavy metals) in compost by using natural zeolite and other structural materials such as ashes.

The aim of our investigation is to process fermented distillers grains into a stable form, and preserve nutrients and reduce nitrogen and carbon losses estimated in a complex manner according to the elementary composition of compost and emission values of gaseous pollutants. It is very important that the process of composting takes places rapidly in a newly designed dynamic composting unit taking account of the optimum compost production term and producing a high quality organic fertilizer.

Methodology

In order to carry out investigations of waste composting, a low-capacity unit for organic waste composting was designed. Composting investigations were conducted in 2008-10 in the Vilnius Gediminas Technical University (VGTU) Laboratory of Environmental Technologies that has composting unit with gas treatment devices (bio-filter). Gaseous pollutant emissions from composted waste were

determined on the basis of standardized methodologies and using the mobile equipment of gaseous pollutant concentration identification of the VGTU Institute of Environmental Protection.

The Components and Qualitative Analysis of Mixtures under Composting

Specimens of fermented waste (distillers grains), fruit-tree (Apple-tree) leaves, bio-fuel ash, and natural zeolite were used for composting. The distillers grains were fermented in a 200 l bio-reactor (filled up to 91%) for a month under anaerobic conditions by maintaining the psychrophilic environment. Prior to the composting experiment zeolite and ashes were dried at 105ºC for 4 hours up to dry mass [26]. Other components of the compost mixture were not additionally treated.

Before starting a composting experiment, the following parameters of the fermented waste, leaves, and bio-fuel ash were determined: humidity, the contents of macro- (C, N, O, Na, Mg, Al, P, S, Cl, K, Ca) and micro-elements (Mn, Zn, Cr, Pb, Cu, Ni), and pH (in compost solution). The chemical composition of the waste and other additives were determined at the Nature Research Centre, other parameters by the VGTU Laboratory of Environment Protection and Labour Conditions.

Preparation of Mixtures for the Experiment

Mixtures containing fermented waste, bio-fuel ash and fruit-tree leaves were composted during the experiment. To reduce gaseous pollutant emissions natural zeolite (0.5-1.00 mm fraction) was used. Ashes were used with the aim of increasing carbon content and at the same time obtaining the optimum carbon-to-nitrogen ratio in the mixtures being composted [28, 29]. The average mass of one mixture reached 10±0.2 kg.

Conformity of all the aforementioned factors is of utmost importance for fermented waste composting and prior to composting, therefore, the calculations of carbon/nitrogen (C/N) ratio and humidity must be carried out [30]. This work used the Compost Calc software [31] of the company Green Mountain Technology, which selected the optimum composition for the mixture under composting at the C/N ratio being from 20/l to 40/l [6]. In order to obtain the required humidity of composted mixtures (40-65%) distilled water was added to the compost, depending on its need [6, 26].

The carbon/nitrogen ratio in the composted mixture was determined according to formula 1 [30]:

$$R = \frac{Q_1(C_1 \times (100 - M_1)) + Q_2(C_2 \times (100 - M_2)) + ...}{Q_1(N_1 \times (100 - M_1)) + Q_2(N_2 \times (100 - M_2)) + ...} \tag{1}$$

...where: $R = C/N$ ratio in the composted mixture; Q_n – mass of component n; C_n – carbon content (%) in component n; N_n – nitrogen content (%) in component n, M_n – humidity of component n (%).

To obtain the optimum ratio R of the composted mixture, it is necessary to evaluate the masses of the mixture's components. For this purpose, Formula 2 is used when the composted mixture has two components:

$$Q_2 = \frac{Q_1 \times N_1 \times \left(R - \frac{C_1}{N_1}\right) \times (100 - M_1)}{N_2 \times \left(\frac{C_2}{N_2} - R\right) \times (100 - M_2)} \tag{2}$$

In the case of three components the following formula is applied:

$$Q_3 = \frac{RQ_1N_1(100 - M_1) + RQ_2N_2(100 - M_2)}{C_3(100 - M_3) - RN_3(100 - M_3)} - \frac{Q_1C_1(100 - M_1) + Q_2C_2(100 - M_2)}{C_3(100 - M_3) - RN_3(100 - M_3)} \tag{3}$$

Humidity in the composted mixture is determined according to the formula:

$$M = \frac{Q_1M_1 + Q_2M_2 + Q_3M_3 + ...}{Q_1 + Q_2 + Q_3 + ...} \tag{4}$$

Calculations completed, the optimum compositions of composted mixtures were determined and are presented in Table 1.

Compost supply with air can be calculated according to the formula:

$$V = 1.2 \cdot M \tag{5}$$

...where: V – supplied air amount $m^3/kg \cdot h$; 1,2 – air amount necessary to maintain aeration of 1 kg compost; M – mass of dry compost mixture, kg.

The minimum duration of the composting process is deemed to be 21 days, the optimum is 30 days.

Compost mixing frequency is determined as follows:

$$S = 4.8075 \exp(0.0896 \cdot r) \tag{6}$$

...where: S – compost mixing frequency, rev/min; r – the layer of composted mixture in the drum, cm.

For example, when a compost layer in the drum is 10 cm its rotation frequency reaches 10 rev/min.

Mixture Composting and Compost Analysis

Composting experiments were carried out from December 2008 until January 2010.

Investigations were performed in a laboratory composting unit (Fig. 1). The total volume of a composting drum to which the mixtures of fermented wastes with additives were manually supplied amounts to 46 l. The composted mixtures were aerated 4 times a day by continuously blowing air for 10 min and rotating the composting unit's drum. The average air supply speed was from 0.2 to 0.5 m/s.

Table 1. Composition of compost mixtures.

Number	Sample/compost mixture	Part of fermented grain in compost, %	Part of bio-fuel ash in compost, %	Part of zeolite in compost, %	Part of leaf in compost, %
1.	K	30	0	0	70
2.	C	10	0	20	70
3.	C+P	10	10	10	70

The average drum rotation speed was 10 rev/min. The average mixture composting span was 4 weeks [32].

When the experiment was completed and the total mass of the composted mixture was removed, the composting unit was washed and cleaned.

Samples of the composted mixtures for the chemical analysis were taken every week from the beginning of composting. The mass of one sample amounted to at least 100 g. Before chemical-analytical investigations, the mixture samples were dried at a temperature no higher than 30ºC and zeolite grains were removed [33]. The samples dried up to the dry mass were crushed in a mortar and sieved through a 2 mm sieve. The content of macro-elements (C, N, Ca, P, K, S, Mg, Na), compost humidity, and pH in the specimens were determined using standardized methodologies [34-36].
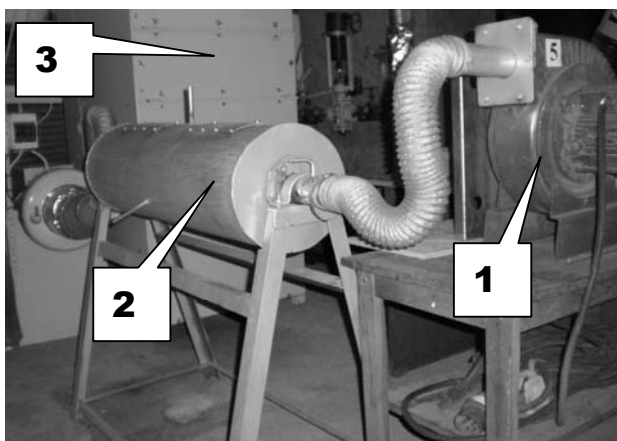

Fig. 1. Composting equipment with air supply and release/treatment systems: 1 – air supply duct with ventilator, 2 – composting cylinder, 3 – biofilter.

Investigation of Gas Emitted during Composting

VOC, H_2S, and methane (CH_4) contents in gas were measured every second day of the experiment since the very beginning thereof. The content of ammonia (NH_3) was automatically recalculated using the coefficients from VOC concentrations (according to MiniRae's description). Concentrations of gaseous components were determined with the mobile laboratory equipment of the Institute of Environmental Protection (methane gas analyzer Alter MG-7, volatile organic compound analyzer MiniRae).

Statistical Evaluation of Research Results

Errors of single-time measurements were taken into account according to the accuracy of the measurement instrument or method as given in the technical specification or standardized methodology [37, 38]. Research results were represented using MS Office package Excel software [39]. For the evaluation of errors of non-stationary processes the software package Statistica using the Kolmogorov-Smirnov coincidence test was applied. It is assumed that the distribution analyzed in the investigation significantly differs from the normal one if the obtained p value is lower than the established level of significance (generally – 0.05) [40]. The regression analysis was carried out according to [41].

Results

The elementary composition of the main components used for composting is presented in Fig. 2. As determined during investigations, the content of nitrogen in fermented
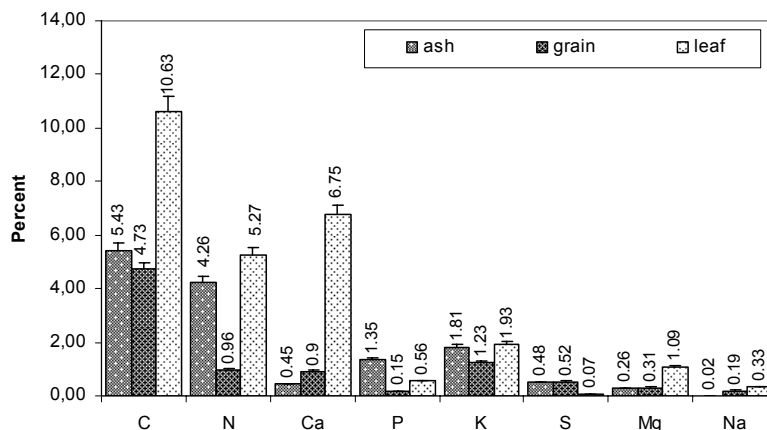

Fig. 2. Elemental composition of grain substrate, biofuel ashes, and zeolite (the ratio of C in ashes and grain is 10:1 by volume).

distillers grain substrate is sufficiently large and reaches 4.26%. In the meantime, other fermented wastes always contain less nitrogen – even half [42]. This substrate also contains fair amounts of macro-elements such as K (1.81%), P (1.35%), S (0.48%), Ca (0.45%), and Mg (0.26%), which are necessary for plants [6]. As determined by scientists [43], the content of the same macro-elements in artificial fertilizers also vary in a similar range.

Comparison of the elementary composition of bio-fuel ash is shown in Fig. 2 and research results of other authors [19, 44] when the average contents of Ca, Mg, K, P, and S in bio-fuel ash account for 10-30%, 1-3%, 2.5-6%, 0.1-1.5%, and 0.1-1.5%, respectively, did not show any significant differences.

Compost produced from leaves is a valuable fertilizer containing much calcium, potassium, magnesium, and sodium [45]. Apple-tree leaves also contain large amounts of nitrogen, which varies in a range of 1-3% [46]. The content of nitrogen determined in the leaves researched was 0.96%, which is by 4.4 and 5.5 times less than in distillers grains and bio-fuel ash, respectively.

The results of compost humidity and pH analysis are shown in Fig. 3, confirming the assumption that the additives of natural zeolite and bio-fuel ash had a considerable influence on the processes of composting: they neutralized compost acidity, and improved its structure and water regime. These processes were analyzed by the scientists [14, 44] who investigated the influence of bio-fuel ash on soils.

As determined by Y. Watanebe [47], one gram of cliptolinolite can absorb around 2.2 g of nitrogen in the form of ammonium. The efficiency of the sorbent *ZeoVit* as regards sewage sludge management has been widely researched in Lithuania [14]. As determined by foreign scientists [25-27], the use of structural additives such as lime and ash, for composting helps to efficiently stabilize biodegradable wastes containing large amounts of biogenic substances as well as those polluted with heavy metals.

Distillers grains are distinguished by an acid medium with their pH being around 3.5-4 [2]. After anaerobic fermentation in the bio-reactor the pH of distillers grains used for composting reached 3.7. Upon adding leaves, the compost alkalized (pH=4.2), while after zeolite insertion pH reached 5.8.
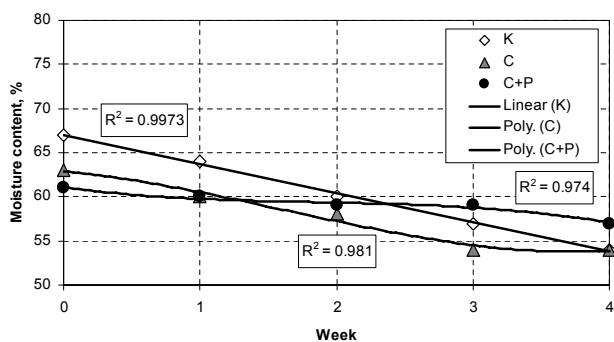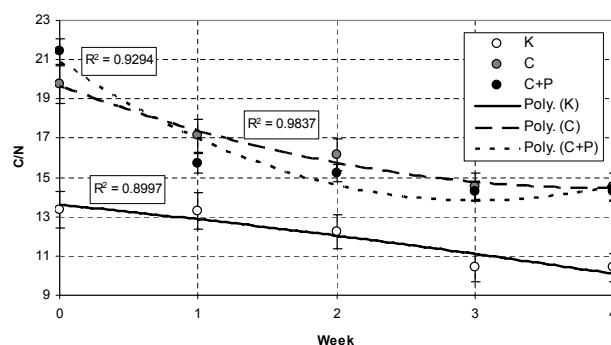

Fig. 4. Total organic carbon and nitrogen (C/N) differences in composted mixtures (K – control specimen, C – compost with zeolite, C+P – compost with zeolite and ash).

As determined by scientists, the increase of zeolite's absorptive capacity NH_4^+ is linear, i.e. when pH=4 nitrogen losses from 1 kg manure are nearly 1.5 times lesser than when pH=7 [48]. L. Charerntanyarak [49] notes that pH=9.5 must be achieved for chemical elements to stabilize. It has been proven by investigations that this limit was not achieved, but during the first week of composting the pH of compost with additives was above 9. Consequently, it can be stated that during that period the thermal regime of compost was intensely changing due to exothermal reactions [50]. At the time the exothermal absorption takes place in the majority of cases the isobar has one or several peaks that must be taken into account when analyzing the intensities of gaseous pollutant emissions [21].

Humidity regimes of distillers grain composting with and without additives showed big differences (Fig. 4). Humidity from grain composted with leaves was lost much more rapidly according to the linear dependence (R^2=0.9973). In the meantime, the porosity and chemical activity of additives (sorbents) used for the investigation allow reducing humidity losses during composting by up to 3.25 times.

Investigations revealed nitrogen and carbon alternation tendencies during composting which lasted for 4 weeks. They allow determining the influence of composting additives on this process. It was noticed that the composting process of control (K) samples had been slow, which is described by an insignificant change in the C/N ratio (Fig. 5).

Upon investigation completion the content of stable macro-elements in composted mixtures increases, i.e. compost mineralization takes place [14, 27], which happens mainly due to humidity evaporation (Fig. 4). This once again proves that composting with additives significantly increases the quality of compost by enriching it with nutrients [51]. When biochemical reactions take place during the process of composting, smaller or bigger losses of carbon and nitrogen are incurred (Fig. 5). The carbon content identified in compost specimens after 4 weeks of composting reached 40.49% (using zeolite alone) and 41.10% (using zeolite and bio-fuel ash). In the meantime, the content of nitrogen fell by 1.02 times when distillers grains were composted with the additive of zeolite, and by around 2.1 times

Fig. 3. Moisture in different composted mixtures (K – control specimen, C – compost with zeolite, C+P – compost with zeolite and ash).
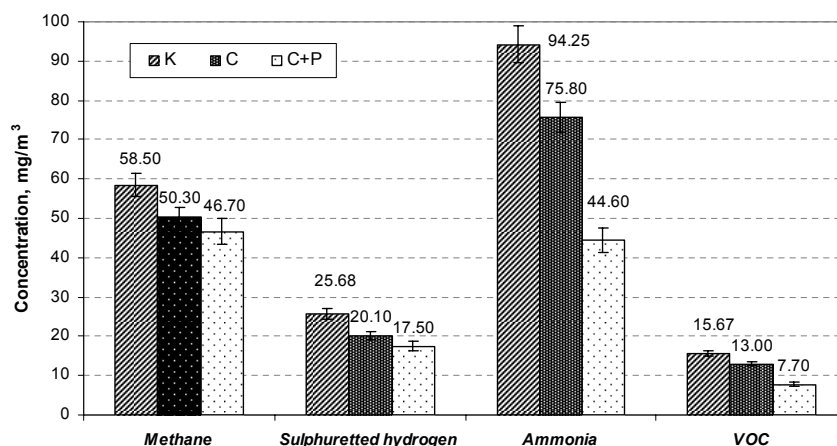
Fig. 5. Average concentrations of gaseous pollutants from 1 kg compost biomass that were obtained when 4 weeks composting (K – control specimen, C – compost with zeolite, C+P – compost with zeolite and ash).

using zeolite and bio-fuel ash. As investigation results show, zeolite efficiently reduces nitrogen loss by 65.6% when used as an additive in composting compared to the control test, and by 84.0% using a mixture of zeolite and bio-fuel.

In the meantime the decrease of carbon content compared to the content identified during control test, in composted mixtures with zeolite and a mixture of bio-fuel and zeolite, reached 20% and 51%, respectively. Naturally, when an organic material is decaying in the process of composting, the content of carbon, chemical energy, proteins and water decrease. It is assumed that the volume of substrate decreases 50% at the end of composting compared to the beginning [32]. As data of Lithuanian scientists' research show, the content of carbon in active surplus sludge substrate of the model test (sludge composting) halved and that of nitrogen decreased by 3.5 times during 10 months [14].

Volatile organic compounds (VOC) are widely spread pollutants generated during various industrial processes. Compost generating greenhouse gas emissions is not an exception.

With the aim of estimating the share of nitrogen lost through compost gas, the investigations of gaseous pollutant emissions were carried out and their results are presented in Fig. 5. As determined by D. B. Lee [52] from National Honam Agricultural Experiment Station (RDA, Iksan (Korea Republic)), nitrogen is mainly lost in the form of ammonia when the C/N ratio is below 15. This is proven by the obtained results of control mixture (K) composting when the concentration of ammonia reaches 160 mg/m³.

When analyzing the influence of zeolite on ammonia emissions from biodegradable waste, Lee determined that the emission of this gas could be reduced up to 70% upon insertion of 5% zeolite. Our investigations show that a 10% additive of zeolite reduced NH_3 emissions from fermented distillers grain compost waste up to 24.3% [52]. In the meantime, when applying bio-fuel ash and zeolite, the efficiency of NH_3 emission suppression more than doubled compared to the control test.

The concentrations of gaseous pollutants emitted from one kg of compost per day during the process of composting were evaluated according to the entire composting process (from the beginning to the end of composting) by using the method of area calculation.

As the performed investigations (Fig. 5) of distillers grain composting show, 1 kg of grain compost mixed with apple-tree leaves, on average, produced 94.25 mg/m³ NH_4. When additives zeolite and zeolite with ash were used, this amount decreased by up to 1.2 and 2.11 times, respectively. The presented data allow an assumption that zeolite blocks a too-high propagation activity of the bacteria degrading organic nitrogen, which slows down the decay of protein organic material, resulting in a lower content of ammonia emission per unit of time [33]. This creates favourable conditions for better use of ammonia with other organisms and inorganic sorbents. Thus, the emission of free ammonia decreases.

When a mixture of distillers grain and leaves was composted, daily emissions, on average, were as follows: CH_4 – 58.5 mg/m³, H_2S – 25.68 mg/m³, and VOC – 15.67 mg/m³. It is determined that the use of natural zeolite reduced CH_4 emissions by 1.16 times and H_2S emissions by 1.28 times, and upon using zeolite with ash these differences reached 1.25 and 1.47 times, respectively.

Comparison with anaerobically digested waste when the hydrogen sulphide (H_2S) concentration in biogas is 2000 ppm [4, 52-54] showed low concentrations of this pollutant in compost gas during composting (up to 40 ppm) (Fig. 5).

Comparison of the obtained results of gaseous emissions with the results of other authors is burdened by a technical problem. Results presented in scientific literature [55-57] are in many cases given in different units, e.g. mg/m² or g/m² units are often used in the evaluation of gaseous pollutant emissions during waste composting in piles. This is why the interpretation of the obtained results is still problematic, which leads to the need of standardizing the methodologies for determining the concentrations of gaseous pollutants emitted during composting.

Conclusions

1. The carbon content identified in compost specimens after 4 weeks of composting reached 40.49% (using zeolite alone) and 41.10% (using zeolite and bio-fuel ash). In the meantime the content of nitrogen fell by 1.02 times when distillers grains were composted with the additive of zeolite, and by around 2.1 times upon using zeolite and bio-fuel ash.

2. As investigation results show, zeolite efficiently reduces nitrogen loss, 65.6%, when it is used as an additive in composting compared to the control test, and by 84.0% upon using a mixture of zeolite and bio-fuel ash.

3. In the meantime, the decrease of carbon content compared to the content identified during control test, in composted mixtures with zeolite and a mixture of bio-fuel ash and zeolite, reached 20% and 51%, respectively.

4. The investigations show that 1 kg of grain compost mixed up with apple-tree leaves, on average, produced 94.25 mg/m^3 NH$_4$. When additives, zeolite, and zeolite with ash were used, this amount decreased by up to 1.2 and 2.11 times, respectively.

5. As investigation results show, when a mixture of distillers grain and leaves was composted, daily emissions, on average, were 58.5 mg/m^3 of CH$_4$, 25.68 mg/m^3 of H$_2$S, and 15.67 mg/m^3 of VOC.

6. It is determined that the use of natural zeolite reduced CH$_4$ emissions by 1.16 times and H$_2$S emissions by 1.28 times, and upon using zeolite with ash these differences reached 1.25 and 1.47 times, respectively.

Acknowledgements

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