The neutralization of organic waste in processes such as composting is related to the emission of malodorous compounds [Sówka et al. 2014]. They include not only the compounds comprised in waste but also biogenic odorants, forming during the first stage of composting and putrefaction (ammonia, hydrogen sulphide, skatole, mercaptans), substances specific for the composting process (aldehydes, geosmin, limonene), transitional substances forming in the process of anaerobic-aerobic transformations (organic acids), forming in pyrolysis and auto-oxidation [Sówka et al. 2014, Frederickson et al. 2013, Kwarciak-Kozłowska and Bańka 2014]. The emission of H₂S or mercaptans can be relatively easily reduced by conducting the process in a proper way (aeration, shovelling heaps). However, there is no possibility to prevent the formation of organic acids or aldehydes.

Owing to their character and low odour detection threshold, there is often a need for a hundred percent elimination of the contaminants from waste gases. One of alternative methods of treating the waste gases from malodorous contaminants, occurring in low concentrations in post-process air, is the biofiltration method. Most often, the method uses an organic filtration material. However, this method of air purification is still developing; therefore, there is a search for new sorbents among mineral materials, which would be an alternative for organic sorbents. The article presents the research into the application of aluminosilicate sorbents, including halloysite, for deodorization of the gases emitted from the processes of composting municipal waste. The semi-technical scale research was conducted for several weeks in a municipal waste composting plant, passing real gases through two biofilters filled with mineral sorbents. In spite of the fact that some problems occurred and the research cycle was not completed, the experiment proved that halloysite removes odours to a much higher extent than the other examined aluminosilicate sorbent. While the VOCs reduction on a bed with halloysite was 88%, the reduction on a bed with a second aluminosilicate reached 35%. The process conditions were very unstable; therefore, the efficiency of the VOCs removal process varied widely. However, halloysite has always been a better sorbent than the other aluminosilicate.

Keywords: Halloysite, odours, composting, biofiltration, aluminosilicates, mineral sorbents
occurring in low concentrations in the post-process air. Its advantage, in addition to relatively low investment and operating costs [Miller et al. 2018], is the fact that this process is practically waste-free. It causes real elimination of contaminants, and not only a change in the place or the form of their occurrence, at the same time not generating extra waste. This makes gas biofiltration a method currently preferred all over the world [Wysocka et al. 2019].

The biofiltration process consists in the slow flow of a contaminated gas through the layer of a sorption material inhabited by the microorganisms specially matched to the type of contaminants. In order to maintain high effectiveness of the process, optimum conditions of gas, bed and operating parameters must be kept. Contaminants diffuse to the liquid coat covering the sorbent, so-called bio-layer or bio-film where, by means of microorganisms and their enzymes, they are decomposed to carbon dioxide and water and the substances are discharged into the atmosphere as harmless and non-toxic [Wierzbińska 2006, Wierzbińska and Modzelewski 2015, Wieczorek 2010].

A biofilter consists of the housing and the layer of a filtration material which fills it, and which is inhabited by the microorganisms adapted to the decomposition of a given type of contaminants. The shape of the biofilter may differ depending on the spatial capabilities and the scale of use [Wierzbińska 2017, Marada and Vaverková 2010]. One of key parameters determining the effectiveness of the gas treatment process is the type of the applied bed, that is, the filtration material. Therefore, there is a need to look for various sorption materials to increase the effectiveness of the deodorization process.

The experiment was conducted at the semi-technical scale in the composting plant, under real difficult conditions. An innovative aspect was the use of mineral sorbents to remove VOCs from the gases emitted from the composting processes. There are no literature reports on similar studies; therefore, the results cannot be compared with other researchers.

**METHODOLOGY**

The research was conducted at the semi-technical scale in the composting plant, under real conditions, passing malodorous gases through the test installation. The conditions were very difficult, because process parameters were very variable.

The research included the observation and assessment of the odour removal process from the waste gases emitted from a composting plant of municipal waste, with the use of two types of sorbents. Two types of mineral sorbents should be used for the research: halloysite – H and aluminosilicate – AS with average granulation of 4-10 mm. The experiment was conducted at the semi-technical scale in the composting plant, under real conditions, passing malodorous gases through the test installation presented in Figure 1.
The installation consists of two biofilters with the height of 1,500 mm and the diameter of 270 mm, made of plastic resistant to chemical and thermal factors, closed from the top with tight lids provided with half-inch tubes and stubs for sampling. At the height of 50 mm from the biofilter bottom stubs are assembled which enable to discharge leachates from beds. Both biofilters were properly filled with sorbents suspended on steel meshes at a height of 200 mm from the bottom of the biofilters. The height of the beds was 1,150 mm. Each biofilter was provided with measurement stubs on the inlet and the outlet. The gases fed onto the biofilters were first driven onto the scrubber in which, owing to the absorption processes in $\text{H}_2\text{SO}_4$, the removal of amine compounds took place. Thus, the gases entering the biofilters were pre-treated from ammonia, among others, and moistened. The parameters of the gas flow through the biofilters were set in such a way that the gas retention time in the bed was 50 seconds. Upstream of the biofilters gasometers were installed, in which the meters were read each time during the sample taking.

On the day when the biofilters were filled up, both beds were inoculated up to a height of 10 from the supporting mesh. The microorganisms used were cultivated on halloysite bed, operating before that at the sewage tank in the composting plant.

The preliminary analysis included a few contaminants characteristic for the process of composting, namely ammonia, hydrogen sulphide, methane, mercaptans, amines, volatile organic compounds (VOCs), and acetic acid. The results are presented in Table 1. The gas samples were taken with the methods matching the applied analytical techniques. Ammonia was taken on the filter made of fiberglass impregnated with sulphuric acid, mercaptans on the filter of fiberglass impregnated with mercury acetate, hydrogen sulphide and VOCs into carbon fibre tubes, methane into Tedlar bags, and to separate primary and secondary aliphatic amines silica gel impregnated with 10% NITC (1-naphthylisothiocyanate) was applied. As there are no studies and emission standards for a composting plant of municipal waste, the analytical method was adjusted to the environmental standards developed by OSHA (Occupational Safety and Health Administration) and NIOSH (National Institute of Occupational Safety and Health). In order to perform markings, the procedures included in the following standards were applied: for ammonia – NIOSH 6015, for mercaptans – NIOSH 2542, for hydrogen sulphide – NIOSH 6013, for amines – NIOSH 2540 and VOCs – OSHA 07 [Eller and Cassinelli 1994] [Ammonia: Method 6015 1994] [Mercaptans, Methyl-, Ethyl-, And N-Butyl-: Method 2542 1994] [Hydrogen Sulfide: Method 6013 1994] [Diethylenetriamine, Ethylenediamine, Triethyleneenetetramine: Method 2540 1994].

As VOCs were of the highest concentration of the pollutants subjected to assessment, further examination of the samples was focused on this group of analytes.

The legal basis was PN-Z-04008–7: 2002 / Az1: 2004 Air purity protection – Sampling – Principles taking air samples in the work environment and interpreting the results.
The method involved the adsorption of vapours of volatile organic compounds on activated carbon, desorption with a mixture of 1% methanol in disulphide carbon and chromatographic analysis (GC / FID) of the obtained solution.

An GC/FID Perkin Elmer AytoSystem XL gas chromatograph with a HP-1 column (100 m × 0.25 mm i.d.), coated by a 0.50 μm crosslinked methyl siloxane film was utilized for the separation of volatile organic compounds occurring simultaneously in the tested air.

The experimental conditions were as follows:
- Carrier gas – helium with flow rate 1.5 cm$^3$/min
- Column temperature program:
  - initial temperature 40°C for 5 min
  - temperature rise 5°C / min up to 100°C
  - intermediate temperature 100°C held for 5 min
  - temperature rise 10°C / min up to 250°C
  - final temperature 250°C held for 3 min
- Dispenser temperature 250°C
- FID temperature 280°C
- hydrogen volume stream 40 cm$^3$ / min
- air flow rate 400 cm$^3$ / min
- 100 : 1 sample divider [Makhniashvili and Kowalska 2007].

### RESULTS

Table 1. The comparison of preliminary measurement results

| Contaminants          | Sample 1   | Sample 2   | Sample 3   | Sample 4   |
|-----------------------|------------|------------|------------|------------|
| Ammonia [mg/m$^3$]    | <0.005     | <0.005     | <0.005     | <0.005     |
| Hydrogen sulphide [mg/m$^3$] | <0.225     | <0.225     | <0.225     | <0.225     |
| Methane [%v/v]        | 0.01       | 1.0        | 0.01       | 0.3        |
| Mercaptans [mg/m$^3$] | <0.03      | <0.03      | <0.03      | <0.03      |
| Aliphatic amines [mg/m$^3$] | 0.30        | 0.26       | 0.28       | 0.56       |
| VOCs [mg/m$^3$]       | 114.8      | 3.4        | 104.1      | 600.2      |
| Acetic acid [mg/m$^3$] | 0.8        | 20.0       | 24.2       | 2.4        |

The samples are numbered according to the numbers of stub ends presented in Figure 1. Moreover, an analysis was conducted for the presence of aldehydes in sample 4, where the presence of acetaldehyde was proven. The analysis of the acetic acid concentration in the samples was carried out to determine pH.

As it results from Table 1, we mainly deal with the oxygen processes here, which is proven by the lack of hydrogen sulphide and mercaptans and non-quantifiable concentrations of methane. On the other hand, in the studied gas there are amines, organic acids and volatile organic compounds (VOCs) in high concentrations.

The analysis results are presented in the source form (Figures 2–5) and converted into the sampling conditions (Tables 3–7). The samples were taken upstream of the scrubber (sample 4), downstream of the scrubber/upstream of the biofilters (sample 1) and downstream of halloysite biofilter – H (sample 2) and aluminosilicate biofilter – AS (sample 3).

Table 2 presents the concentrations of aliphatic and aromatic hydrocarbons in the studied samples. The sum of hydrocarbons in the studied samples converted into the amount of the air taken (VOCs) and the conditions of sampling are presented in Tables 3–7.

### Analysis of the results

As it arises from the analysis of the results listed in Table 2, after a one week period of the operation of the experimental installation for the deodorization of waste gases, the concentration of VOCs in the gas after the biofiltration process increased substantially. The concentrations of aliphatic hydrocarbons in the samples taken increased after passing through the H bed by 70%, and by 140% after the AS bed. Likewise, after the H sorbent, the concentrations of aromatic hydrocarbons increased by 93%, and after AS – by 220%. Thus, the concentrations of VOCs increased by 1.7 times after H and 26 times after AS (Figure 2). In that stage, there were no microbiological reactions yet, which were supposed to be the complementation of the deodorization process. It was proven by the lack of CO$_2$ in the exhaust gas, which was not considered in the tables. The total decomposition of organic compounds to H$_2$O and CO$_2$ occurs during biological transformations.

After the next few days, the concentrations of VOCs were reduced on both sorbents but on
the outlet of the biofilters, the concentration was lower by 22% in the case of halloysite and by 15% in the case of aluminosilicate. In comparison with the concentrations on the inlet to the installation (upstream of the scrubber), the concentrations of VOCs were lower by 91% and 57%, respectively. The temperature of the gas on the outlet of the installation with the H bed was 6°C lower than downstream of the bed with AS. The pH was above 6.

After two weeks of the operation of the research installation at the semi-technical scale, both biofilters started to perform their expected function, but the reduction of contaminants took place to a various extent. The reason for that were very unstable conditions before the installation, that is still before absorption in H<sub>2</sub>SO<sub>4</sub> (upstream of the scrubber). The concentrations of VOCs downstream of the biofilters, in comparison with concentrations on the installation inlet, dropped by 88% downstream of the halloysite bed and by 35% downstream of the bed containing aluminosilicate, but the concentration downstream of the AS sorbent was more than 5 times higher than downstream of the H sorbent. The pH of the gas fluctuated around 5.67–5.90, whereas the leachate pH was above 7. The humidity of the gas downstream of the H bed was 74%, and downstream of the AS bed, it was 96% (series III).
Figure 3. The comparison of chromatograms showing decomposition of VOCs in the industrial gas upstream of the biofilters (1) and downstream of the H biofilter (2) and the AS biofilter (3) (2nd series)

After 18 days of research, the level of the removal of VOCs on H sorbent was 65%, and the concentration of contaminants after deodorization on the AS sorbent increased by 57% (Figure 4). In comparison with the concentration of VOCs which was examined upstream of the scrubber, it dropped by 26% downstream of the H bed, but it increased by 127% downstream of the AS bed. The temperature of gases on the outlet from the H bed and the AS bed were similar and amounted to about 23°C. The humidity of gases downstream of the H bed was 87%, and downstream of the AS bed it was 90.9%, which would indicate more intensive absorption of humidity by the halloysite sorbent (series IV) (Table 5).

Series V showed an analogous trend. There was a decrease in the concentration of VOCs by 87% on the H sorbent and an increase by 24% on the AS sorbent (Figure 5). The input concentration was more than twice higher than during the previous gas extraction. The pH of the gas downstream of the beds was relatively low and amounted to 5.92 and 5.64, respectively, with the pH of the leachate slightly below 7. The temperature of the gases on the outlet from the installation differed between the beds by almost 6°C. The humidity of gases was 66% downstream of the H bed and 72% downstream of the AS bed; therefore, it was lower than the required one and probably resulted from the process of water sorption on the sorbents (Table 6).
After a few days, another analysis of the samples of the air contaminated with Volatile Organic Compounds was conducted. Just like before, halloysite showed better sorption. It reduced the concentrations of organic contaminants by 82%, whereas on the outlet of the gas from the biofilter with the aluminosilicate sorbent, the concentrations increased by 11%. The pH of the gas and leachate was below 6, and temperatures of the gas downstream of the H and AS beds were 28 and 25°C, respectively. Gas humidity on the outlet of the installation was around 97% (Table 7).

During the experiment, the temperature of the gas on the outlet from the H biofilter was in the range of 17–28°C, whereas downstream of the AS biofilter, it was 23–29°C (Tables 3–7). The pH of the gas downstream of the H bed changed in the range from 5.55–6.22, and downstream of the AS bed, it was 5.51–6.04 (Tables 3–7). What results from it is that the conditions upstream of the scrubber and the biofilters were very unstable, and the concentrations of odours were changeable, which is proven by the charts presented in Figures 2–5. One can observe that in spite of the fact that both sorbents operated under the same conditions, the H sorbent performed its function much better. In the majority of cases, it reduced the concentrations of selected odorants, deviating substantially from the AS sorbent. The H sorbent turned out to be a good sorbent for VOCs, and the AS sorbent did not prove to be efficient in removing VOCs from gases. The changeability of VOCs concentrations (the selected results-representative series) is also shown in Figure 6.
Figure 5. The comparison of chromatograms showing decomposition of VOCs in the industrial gas upstream of biofilters (1) and downstream of the H biofilter (2) and the AS biofilter (3) (5th series)

As the results collected over the few weeks of the research with the use of two mineral biofilters: halloysite and aluminosilicate show, the period was characterised by high instability of the operation of sorbents. It is characteristic for the period of the development of biofilters, when mainly the process of contaminant sorption on the sorption material takes place. After a few weeks from inoculation of the bed, the deodorization process begins to base on the microbiological processes, owing to the organisms which had time to reproduce and settle the whole filtration material during the development of the biofilter. The experiment, however, was interrupted before the process of removing mineral contaminants from the industrial gas stabilized due to the problems which occurred during the operation of the testing biofilters. The most serious was excessive humidity of the gas flowing to the biofilters. There was a malodorous brown liquid flowing to the biofilters from the tubes transporting the process air. Settling on the bottom of the biofilters under the grate on which the bed was suspended, the liquid was an additional source of odours, which disturbed the experiment. It required making siphons enabling to discharge the liquid before entering the reactors. It was also necessary to raise the biofilters to such a height that would allow free drainage of the excess of humidity. Moreover, flooding of bellows gasometers, from which the malodorous brown
liquid was poured out as early as after a week of the operation (Photo 1), has occurred.

An additional difficulty was relatively low pressure of the gas in the cables supplying the gas to biofilters. Initially, it was 300 Pa, decreasing after a week to about 100 Pa, and after two weeks even to 50 Pa. The drop was probably caused by excessive humidity in the bed, where, as a result

Table 2. The comparison of the concentrations of hydrocarbons in the studied samples

| Series | Sample no. | Concentration in the sample [µg] | Aliphatic hydrocarbons | Aromatic hydrocarbons |
|--------|------------|---------------------------------|------------------------|-----------------------|
| 1      | 1          | 421.03                          | 92.54                  |                       |
| 2      | 2          | 715.68                          | 178.64                 |                       |
| 3      | 3          | 1039.30                         | 294.21                 |                       |
| 4      | 4          | 580.25                          | 238.83                 |                       |
| 1      | 1          | 1090.02                         | 381.09                 |                       |
| 2      | 1          | 135.70                          | 135.08                 |                       |
| 3      | 1          | 1161.95                         | 91.70                  |                       |
| 4      | 1          | 3545.17                         | 2228.63                |                       |
| 1      | 2          | 1891.98                         | 197.41                 |                       |
| 2      | 1          | 157.72                          | 116.07                 |                       |
| 3      | 1          | 1341.77                         | 182.51                 |                       |
| 4      | 1          | 1986.03                         | 371.87                 |                       |
| 1      | 2          | 739.18                          | 230.67                 |                       |
| 2      | 2          | 186.46                          | 161.00                 |                       |
| 3      | 2          | 1351.33                         | 175.31                 |                       |
| 4      | 2          | 324.30                          | 141.57                 |                       |
| 1      | 3          | 1291.54                         | 609.21                 |                       |
| 2      | 3          | 133.95                          | 120.18                 |                       |
| 3      | 3          | 1634.10                         | 721.21                 |                       |
| 4      | 3          | 3920.82                         | 798.12                 |                       |
| 1      | 4          | 1173.38                         | 529.93                 |                       |
| 2      | 4          | 123.28                          | 109.37                 |                       |
| 3      | 4          | 1729.02                         | 618.91                 |                       |
| 4      | 4          | 2219.03                         | 876.20                 |                       |

Table 3. The selected results of analyses – 2nd series

| Specification | Sample no. 4 upstream of the scrubber | Sample no. 1 downstream of the scrubber/ upstream of biofilters | Sample no. 2 downstream of H biofilter | Sample no. 3 downstream of AS biofilter |
|---------------|---------------------------------------|----------------------------------------------------------------|---------------------------------------|---------------------------------------|
| VOCs [mg/m³]  | 288.69                                | 147.11                                                           | 27.08                                 | 125.37                                |
| pH of gas     | 7.16                                  | 6.40                                                             | 5.55                                  | 5.92                                  |
| pH of leachate| 7.57                                  | 6.10                                                             | 6.83                                  | 6.43                                  |
| Temperature °C| -                                     | -                                                                | 23.0                                  | 29.0                                  |

Table 4. The selected results of analyses – 3rd series

| Specification | Sample no. 4 upstream of the scrubber | Sample no. 1 downstream of the scrubber/ upstream of biofilters | Sample no. 2 downstream of H biofilter | Sample no. 3 downstream of AS biofilter |
|---------------|---------------------------------------|----------------------------------------------------------------|---------------------------------------|---------------------------------------|
| VOCs [mg/m³]  | 235.79                                | 163.03                                                           | 27.38                                 | 152.43                                |
| pH of gas     | 5.80                                  | 6.73                                                             | 5.67                                  | 5.90                                  |
| pH of leachate| 8.06                                  | 6.26                                                             | 7.84                                  | 7.36                                  |
| Temperature °C| -                                     | -                                                                | 17.0                                  | 23.5                                  |
| Humidity [%]  | -                                     | -                                                                | 74.0                                  | 96.3                                  |

Photo 1. Malodorous liquid poured out of the gasometer
Table 5. The selected results of analyses – 4th series

| Specification | Sample no. 4 upstream of the scrubber | Sample no. 1 downstream of the scrubber/ upstream of biofilters | Sample no. 2 downstream of H biofilter | Sample no. 3 downstream of AS biofilter |
|---------------|--------------------------------------|---------------------------------------------------------------|---------------------------------------|----------------------------------------|
| VOCs [mg/m³]  | 460.59                               | 96.98                                                         | 34.75                                 | 152.66                                 |
| pH of gas     | 5.56                                 | 5.35                                                          | 6.22                                  | 6.04                                   |
| pH of leachate| 7.42                                 | 6.03                                                          | 6.73                                  | 6.21                                   |
| Temperature [°C] | -                                     | -                                                             | 23.4                                  | 23.8                                   |
| Humidity [%]  | -                                    | -                                                             | 87.0                                  | 90.9                                   |

Table 6. The selected results of analyses – 5th series

| Specification | Sample no. 4 upstream of the scrubber | Sample no. 1 downstream of the scrubber/ upstream of biofilters | Sample no. 2 downstream of H biofilter | Sample no. 3 downstream of AS biofilter |
|---------------|--------------------------------------|---------------------------------------------------------------|---------------------------------------|----------------------------------------|
| VOCs [mg/m³]  | 372.52                               | 190.08                                                        | 25.41                                 | 235.53                                 |
| pH of gas     | 6.08                                 | 5.47                                                          | 5.92                                  | 5.64                                   |
| pH of leachate| 7.79                                 | 6.42                                                          | 6.81                                  | 6.67                                   |
| Temperature [°C] | -                                    | -                                                             | 17.5                                  | 23.0                                   |
| Humidity [%]  | -                                    | -                                                             | 66.0                                  | 72.1                                   |

Table 7. The selected results of analyses – 6th series

| Specification | Sample no. 4 upstream of the scrubber | Sample no. 1 downstream of the scrubber/ upstream of biofilters | Sample no. 2 downstream of H biofilter | Sample no. 3 downstream of AS biofilter |
|---------------|--------------------------------------|---------------------------------------------------------------|---------------------------------------|----------------------------------------|
| VOCs [mg/m³]  | 222.13                               | 174.51                                                        | 31.34                                 | 193.60                                 |
| pH of gas     | 6.10                                 | 5.67                                                          | 5.40                                  | 5.51                                   |
| pH of leachate| 5.72                                 | 5.70                                                          | 5.53                                  | 5.64                                   |
| Temperature [°C] | -                                    | -                                                             | 28.3                                  | 25.0                                   |
| Humidity [%]  | -                                    | -                                                             | 97.0                                  | 97.1                                   |

Figure 6. The selected results of changes in the concentrations of VOCs in the industrial gas after passing through the halloysite and aluminosilicate bed
of the condensation of water vapour, the water filled the pores of the sorbents and resistances of the already insufficient flow increased. Anaerobic zones occurred, where undesired bacterial flora began to develop, probably displacing the aerobic bacteria inoculated during the start of the installation. It would also explain the results of the olfactometric analyses carried out after three weeks of the operation of biofilters, which showed 150 000 ou downstream of the H biofilter, and 300 000 ou downstream of the AS biofilter, while upstream of the biofilters the value was 100 000 ou. It also proves that the dominating source of odours was the fermenting sludge on the bottom of the biofilters. A solution would be to install a pump which would force the flow of gas in the bed, additionally oxygenating the inoculated microflora.

To sum up, it should be noted that in spite of atypical and unstable conditions which took place in the installation while conducting the experiment, the applied sorbents had a tendency to stabilize the efficiency of the deodorization of the gas emitted from the processes of municipal waste composting. It can be assumed that if the conditions had been stable and optimal, both sorbents would prove the high efficiency of odour removal. However, it should be pointed that almost all the obtained results show that halloysite is a better sorbent than aluminosilicate. We do not know if it is also a better medium for the inoculated microflora, because the experiment lasted too short to provide such information.

These results cannot be compared with other researchers, because similar studies have not been described in the literature.

CONCLUSIONS

1. The experiment planned at the semi-technical scale did not allow determining the overall efficiencies of mineral biofilters due to unstable operating conditions and early termination of the experiment.
2. The halloysite sorbent is a mineral material which is potentially more efficient in biofiltration than the aluminosilicate sorbent.
3. In the first week of the research, when the deodorization process consisted only in the sorption of contaminants on the bed and the damping of sorbents did not take place yet, halloysite eliminated Volatile Organic Compounds in almost 100%.
4. Halloysite proved to be relatively effective in removing VOCs, whereas aluminosilicate did not cope with VOCs at all.
5. In that specific composting plant, the odour problem is created by VOCs, acetaldehyde and acetic acid, whereas such contaminants as: ammonia, hydrogen sulphide, methane and mercaptans can be considered negligible.

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