Application of aluminosilicates for mitigation of ammonia and volatile organic compound emissions from poultry manure

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Abstract: Odor mitigation techniques are widely investigated due to the problem of odor nuisance generated by intensive livestock production. The goal of this research was to investigate the use of aluminosilicate sorbents as filter packs in the air scrubber ODOR1, which enables cleaning of air inside the livestock building. The following sorbents were examined: raw halloysite, roasted halloysite, activated halloysite, raw bentonite, roasted bentonite and expanded vermiculite. The experiment was conducted in chambers where poultry manure was placed, the time of air treatment was 24 hours. A manual SPME (solid-phase microextraction) holder with DVB/Carboxen/PDMS fiber was used for extraction of odor compounds, and analyses were carried out using gas chromatography-mass spectrometry. Ammonia concentrations were determined according to Polish standards (Nessler method) using a spectrophotometer. It was found that all examined aluminosilicates had the potential for removal of ammonia as well as 24 volatile compounds emitted from poultry manure. The highest efficiency was noted for activated halloysite (81%) and roasted bentonite (84%) in the case of ammonia and odors, respectively. Despite the limitations of the study, the results showed the effectiveness of the air scrubber packed with aluminosilicates for the reduction of volatile odorous compounds in the air of livestock buildings.

Keywords: poultry manure, ammonia, volatile organic compounds, air scrubber, aluminosilicates

1 Introduction

Odor annoyance, specifically the emission of odorous compounds into the environment, is strongly associated with intensive livestock production. Moreover, public complaints about livestock facilities are mainly related to odorous compounds that are polluting rural locations [1]. The problem of air pollution from industrial farms is not new and has still not been definitively resolved, in spite of the numerous studies that have been undertaken. The main methods of odor prevention and control strategies focus on reducing odor formation, using end-of-pipe techniques and enhancing dispersion [2]. However, the main problem is that livestock odors are a mixture of hundreds of different compounds depending on the types of animals [3-7]. Thus odor prevention techniques should focus on the most significant odor-causing compounds.

Among the methods used to reduce emission of odorous compounds from livestock buildings the following can be distinguished: air scrubbing [8], biofiltration [9], use of manure covers [10], litter amendments and masking agents [11]. Furthermore, the composition of animal excreta is related to the composition of their diet. Therefore supplementation of various types of feed additives such as natural antimicrobial additives and plant-derived essential oils [12], multistrain probiotics [13] or supplemental synthetic amino acids [14] has also been investigated.

To date, the investigated air cleaning systems such as biofilters, scrubbers and biotrickling filters have used wet packing materials in order to purify air from livestock buildings. Although such odor mitigation techniques were relatively effective compared to other methods, it is not possible to use them in naturally ventilated buildings. Moreover, there are some limitations in reducing odor
concentrations because only soluble compounds are removed, which does not include many odorants [15]. Thus, the objective of this research was to investigate the use of aluminosilicate sorbents as filter packs in the air scrubber ODOR1, which enables cleaning of air inside the livestock building.

2 Experimental procedure

2.1 Investigated Sorbents

The following sorbents were examined: raw halloysite (H), roasted halloysite (HR), activated halloysite (HA), raw bentonite (B), roasted bentonite (BR) and expanded vermiculite (EV). The height of the filtering layer was about 8 cm, which corresponded to 1.5 kg (EV) and 12 kg (other aluminosilicates) of examined sorbents due to the different density of materials. The empty bed residence time (EBRT), which equals the sorbent volume/volumetric flow rate, was 0.12 s. Physical-chemical properties of the investigated sorbents were reported previously by Opalinski et al. [16].

2.2 Experimental Site and Procedure

The research was conducted in three identical air-conditioned chambers equipped with the SCADA PRO-2000 system (MikroB S.A., Poland), which enables constant monitoring of temperature and relative humidity of air. Nearly fresh hen manure was collected from commercial farms and was placed in each chamber so as to obtain the surface emission of about 8 m². The air scrubber (Fig. 1) was in the middle of each chamber and was filled with investigated aluminosilicates except the chamber (control), where filter packs were empty. In order to obtain three replicates of each treatment (aluminosilicate), nine series were conducted, two aluminosilicates (one in each chamber) and one control treatment for each series. The time of air purification for each series was 24 hours and at the end two samples of air were taken from each chamber. The chambers were ventilated for 24 hours without air purification between each series and filter packs were filled with a new batch of investigated sorbents. Hen manure was also mechanically mixed to avoid crusting of the emission surface. Taking into account the volume of the container (23 m³) and the filtration efficiency of the air scrubber ODOR1, the air inside was filtered about 40 times per hour which is in agreement with recommended maximum summer ventilation rates reported by Seedorf et al. [17].

2.3 SPME Sampling and Chemical Analysis

A manual SPME holder with StableFlex 50/30 µm DVB/Carboxen/PDMS fiber (Supelco, Bellefonte, PA) was used for extraction of odor compounds from the headspace inside the experimental chambers and the optimal 60 minute sampling time was determined during preliminary experiments. SPME fibers were conditioned for 1 hour in the injection port (270°C) each time before collection of samples. Chromatographic separation was performed using a Restec column (5% diphenyl; 30 m × 250 µm × 0.25 µm) and gas chromatograph coupled with a mass spectrometer (Finnigan Polaris Q type, Thermo Electron, USA). The general run parameters used were as follows: injector 240°C; transfer line 220°C; column, 40°C initial, 2 minute hold, 4°C min⁻¹, 240°C final, 10 minute hold; carrier gas, helium. Mass/charge (m/z) ratio range was set between 33 and 280 amu (atomic mass units). Spectra were collected at 6 scans sec⁻¹ and the electron multiplier voltage was 1200 to 1350 V. The detector was auto-tuned weekly.

Odorous compounds were tentatively indentified on the basis of comparative analysis of the determined mass spectrum and the spectrum from the NIST (National Institute of Standards and Technology) commercial MS library.

Evaluation of air purification effectiveness was made on the basis of the relative reduction value (RRV, %) determined for each tentatively indentified odorous compound. The RRV was given as the mean (of three replicates) and it was calculated according to the formula
reported by Cai et al. [18] as the ratio of the difference between the control and treatment peak area count of the tentatively identified odorous compound to the control.

Moreover, samples of air for assessment of ammonia concentration were collected after the filtration process, in the middle of each chamber, using an AMZ-1 aspirator (Rotametr, Poland). The content of ammonia was determined in duplicate according to Polish standards (Nessler method) using a UV-3100 PC spectrophotometer (VWR International bvba, Leuven, Belgium).

### 2.4 Statistical Analysis

The data was tested for normality (Shapiro-Wilk test) and statistical comparisons between the treatments were done via one-way ANOVA using Statistica version 8.1 (Statistica for Windows, StatSoft Inc., Tulsa, OK). Differences among treatment means were tested for significance using Tukey’s HSD test. Differences were considered significant at P < 0.05.

### 3 Results and discussion

It was found that all examined aluminosilicates had potential for removal of ammonia from the air inside the experimental chambers (Table 1). The concentration of ammonia in the samples of air collected from the control chamber was always significantly higher than 5 in the samples from treatment groups (p < 0.05): 11 and 55 mg kg⁻¹ in the HA treatment and the control respectively. The highest efficiency was noted for activated halloysite (81%) and the lowest for roasted halloysite (36%). Moreover, the differences in the ammonia concentration reduction level between HA or BR treatment and the other examined aluminosilicates were about 40%, and they were statistically confirmed.

A total of 24 volatile compounds were tentatively identified from the headspace of chambers (Table 2). According to results reported previously by Lo et al. [19], 13 of 24 compounds found in this research have a distinct odor, and moreover, five are listed as hazardous air pollutants, i.e. 2-butanone, 1,2-dimethylbenzene, 1,4-dichlorobenzene, phenol and 1-H-indole (USEPA, 2011. Air toxics website, Washington, DC, USA). The results demonstrated that all investigated aluminosilicates had the ability to adsorb the tentatively identified volatile compounds and the mean overall relative reduction value was between 56 and 84% for raw halloysite and roasted bentonite, respectively. The differences in the calculated RRV level between the treatments were generally not statistically significant, though in the case of seven compounds, according to the literature five of which have a distinct odor, the differences were significant (p < 0.05). It was found that the most effective was roasted bentonite (Fig. 2), for which in the case of seven compounds the RRV was above 90%, while for 1H-indole (barnyard/fecal aroma), dimethyl trisulfide (onion/fish aroma) and pyridine (rancid/pungent aroma) it was even 100%. The potential for removal of tentatively identified volatile compounds by investigated aluminosilicates was in the order BR > HR > HA > EV > B > H.

The differences of the potential for removal of ammonia and odors determined for the investigated aluminosilicates were probably the result of different surface properties. The sorption ability of clays is mainly determined by the chemical nature and pore structure. Strong retention of pollutants on the surface of clays is due to the specific interactions including hydrogen bonding, complexation or acid/base interactions. The adsorption centers for ammonia consist of both Lewis and Brønsted centers. Moreover, the specific surface area and surface acidity of the aluminosilicates samples can be greatly increased by acid activation [20,21]. That would explain the best results obtained for activated halloysite and roasted bentonite and reported in the present work.

### Table 1: Effect of aluminosilicates on reduction of ammonia concentration inside the chambers (mean, n=3).

| Number of series | Investigated sorbent | Control | H | HR | HA | B | BR | EV | SEM | P-value |
|-----------------|----------------------|---------|---|----|----|---|----|----|-----|---------|
| I-III           | Ammonia concentration [mg kg⁻¹] | 55a § | 33b | 36b | - | - | - | - | 3.93 | 0.006 |
|                 | Ammonia reduction [%] |        | 41 | 36 | - | - | - | - |       |         |
| IV-VI           | Ammonia concentration [mg kg⁻¹] | 56a | - | - | 11b | 31c | - | - | 6.76 | >0.001 |
|                 | Ammonia reduction [%] |        | - | - | 81 | 45 | - | - |       |         |
| VII-IX          | Ammonia concentration [mg kg⁻¹] | 53a | - | - | - | - | 13b | 31c | 5.81 | >0.001 |
|                 | Ammonia reduction [%] |        | - | - | - | - | 75 | 41 |       |         |

H, raw halloysite; HR, roasted halloysite; HA, activated halloysite; B, raw bentonite; BR, roasted bentonite; EV, expanded vermiculite; §, Values followed by the same lowercase letter are not significantly different at P <0.05 using the Tukey’s HSD test.
Similar effects were reported by Opalinski et al. [22] when aluminosilicates were tested to purify the air inside containers where fresh cow’s manure was placed. The highest reduction level of ammonia content was about 68 and 77% for roasted bentonite and activated halloysite respectively. It was also found that investigated aluminosilicates effectively reduced the concentration of the tentatively identified odors. Moreover, when a prototype of the filtering machine was used in the vivarium for laying hens, the reduction rate of sulphur organic compounds was on the level of 43% and 60%, for halloysite and bentonite respectively [23]. Investigated aluminosilicates were also tested during a 10 day filtration trial and the average ammonia reduction level was 47% for activated halloysite and 35% for roasted bentonite [24]. The application of aluminosilicates as a filter bed for air scrubbers in order to improve the poultry house environment was reported by Koelliker et al. [25]. A simple air scrubber packed with six layers of the clinoptilolite (4.64 kg, granulation 2.35-4.70 mm) removed 15 to 45% of the NH$_3$-N from the air even though the contact time was less than 1 second. Moreover, the investigators and personnel of the poultry house were convinced that the device reduced odor intensity and offensiveness. However, when clinoptilolite was added to the broiler feed (2% wt.) and was also spread, 1.6 kg per m$^2$ of litter surface, during the six-week experiment, the ammonia concentration was higher than in the control and the total mass of NH$_3$ emitted was 50% greater [26]. Nevertheless, Turan et al. [27] reported that expanded vermiculite had the ability to reduce, by about 60%, the content of volatile organic compounds emitted during composting of poultry litter. Furthermore, Cai et al. [18] found that topical application of zeolite to laying hen manure caused a reduction of the total odor measured with the gas chromatography-olfactometry (GC-O) approach by 51 to 67%. Finally, the effectiveness of aluminosilicates investigated in the present research was comparable to the efficiency of other frequently tested methods such as biofiltration. Chen et al. [28] examined a pilot-scale mobile biofilter filled with two types of wood chips in order to reduce odor emissions.

*Figure 2: Comparison of chromatogram between BR treatment and control, numbers are corresponding with tentatively identified compounds in Table 2.*
from a deep-pit swine finishing facility. The results demonstrated that overall average reduction efficiency for treating odor compounds was between 76 and 93%. However, as mentioned before, the biofiltration method is not available inside the livestock building, unlike the air scrubber packed with mineral sorbents that has been tested in the present study.

It is worth noting that spent scrubber sorbents have value as a soil amendment, and thus could be applied to land for final disposal. It was observed that when BR was composted for 100 days with poultry litter, straw and peat, it was toxic neither to earthworms nor to plants. Furthermore, addition of 5 and 10% wt. of BR compost to the test soil had a positive influence on the yield of mustard seeds: about three and six times higher yield respectively compared to the control treatment [29]. Moreover, spent activated halloysite and roasted bentonite, obtained after air filtration, were applied as an additive to granulated iron concentrate used in a cement kiln during clinker production. It was found that the investigated sorbents can be used as a substitute for sodium bentonite, which could be an alternative method for safe final disposal [30].

Although the RRV levels obtained for the investigated sorbents were high, it is worth underlining that the calculated contact time (EBRT) between the air and the aluminosilicates was very short (0.12 seconds). Thus, we assume that the greater height of the filtering layer inside the air scrubber would be profitable in order to obtain higher RRV. Moreover, because the air scrubbing process was examined for only 24 hours, the breakthrough time for each aluminosilicate was not assessed. These facts are noteworthy and need further investigations.

Furthermore, it is very important to characterize volatile odorous compounds that are subjected to air

| Investigated sorbent | Compound name | H | RRV | SD | HR | RRV | SD | HA | RRV | SD | B | RRV | SD | BR | RRV | SD | EV | RRV | SD | SEM | P-value |
|----------------------|---------------|---|-----|----|----|-----|----|----|-----|----|---|-----|----|----|-----|----|----|-----|----|-----|----------|
| 1.                    | Diethyl sulfide | 15.44 | 68 | 9 | 77 | 12 | 86 | 10 | 75 | 8 | 97 | 2 | 67 | 22 | 3.51 | 0.0716 |
| 2.                    | Butanone #, †  | 16.18 | 44 | 12 | 43 | 18 | 45 | 13 | 44 | 21 | 49 | 8 | 37 | 27 | 3.58 | 0.9752 |
| 3.                    | Propanol | 18.72 | 43 | 19 | 62 | 10 | 57 | 14 | 37 | 25 | 57 | 12 | 33 | 22 | 4.53 | 0.3268 |
| 4.                    | Methyl ethyl disulfide | 19.51 | 41 | 34 | 55 | 17 | 37 | 16 | 37 | 17 | 74 | 7 | 52 | 23 | 5.13 | 0.2918 |
| 5.                    | Pentanone # | 20.75 | 68 | 11 | 79 | 12 | 71 | 6 | 66 | 12 | 84 | 6 | 75 | 4 | 2.34 | 0.1995 |
| 6.                    | Diethyl disulfide | 20.94 | 40a$ | 15 | 47 | 17 | 71 | 2 | 53 | 9 | 76b | 7 | 52 | 16 | 3.95 | 0.0241 |
| 7.                    | Heptene | 21.68 | 64 | 9 | 65 | 17 | 64 | 16 | 66 | 6 | 87 | 6 | 63 | 10 | 3.26 | 0.1423 |
| 8.                    | 1,2-Dimethylbenzene #, † | 22.66 | 59a | 12 | 79 | 11 | 79 | 8 | 73 | 6 | 86b | 9 | 65 | 8 | 2.85 | 0.0374 |
| 9.                    | Methyl-2-pentanol | 22.87 | 67 | 8 | 82 | 11 | 72 | 6 | 58 | 9 | 82 | 7 | 72 | 14 | 2.77 | 0.0697 |
| 10.                   | Pentadieninitrile | 23.06 | 70 | 7 | 79 | 11 | 71 | 8 | 63 | 14 | 85 | 8 | 68 | 7 | 2.57 | 0.1068 |
| 11.                   | Limonene # | 23.35 | 66 | 7 | 73 | 4 | 75 | 7 | 64 | 15 | 84a | 9 | 51b | 16 | 3.26 | 0.0333 |
| 12.                   | Pyridine # | 23.88 | 86a | 9 | 100b$ | 0 | 100b | 0 | 100b | 0 | 100b | 0 | 91 | 4 | 1.58 | 0.0012 |
| 13.                   | Pentanal | 24.84 | 57 | 9 | 74 | 13 | 75 | 10 | 61 | 16 | 86 | 12 | 69 | 5 | 3.17 | 0.0788 |
| 14.                   | 2-Methyl-2-heptanone | 25.10 | 56a | 15 | 80 | 11 | 73 | 4 | 57a | 11 | 92b | 6 | 74 | 18 | 3.86 | 0.0176 |
| 15.                   | Octanone # | 25.62 | 4a | 37 | 59 | 15 | 29ac | 22 | 69bc | 16 | 94b | 6 | 55 | 12 | 8.03 | 0.0031 |
| 16.                   | Methyl trisulfide # | 25.85 | 79a | 10 | 88 | 7 | 94 | 4 | 87 | 8 | 100b | 0 | 94 | 1 | 2.04 | 0.0222 |
| 17.                   | 1,4-Dichlorobenzene † | 26.95 | 45 | 22 | 82 | 8 | 72 | 18 | 61 | 13 | 80 | 6 | 65 | 8 | 4.06 | 0.0531 |
| 18.                   | Ethyl-1-hexanol # | 27.85 | 77 | 18 | 95 | 7 | 95 | 8 | 95 | 5 | 99 | 2 | 93 | 4 | 2.49 | 0.1184 |
| 19.                   | Nonanone # | 28.34 | 52a | 28 | 71 | 12 | 74 | 16 | 78 | 12 | 99b | 1 | 70 | 16 | 4.63 | 0.0760 |
| 20.                   | Dimethylphirimidine | 35.91 | 48 | 33 | 77 | 7 | 73 | 14 | 66 | 21 | 96 | 4 | 76 | 11 | 4.93 | 0.0114 |
| 21.                   | Undecanone # | 38.73 | 42 | 30 | 68 | 17 | 59 | 15 | 42 | 17 | 64 | 7 | 50 | 14 | 4.36 | 0.3543 |
| 22.                   | Phenol #, † | 39.05 | 62 | 15 | 77 | 12 | 70 | 23 | 59 | 16 | 97 | 5 | 69 | 11 | 4.20 | 0.0789 |
| 23.                   | 4-Methylphenol #, † | 40.73 | 16 | 45 | 48 | 19 | 36 | 52 | 19 | 29 | 44 | 33 | 37 | 57 | 9.15 | 0.9168 |
| 24.                   | 1H-Indole # | 44.21 | 80 | 14 | 94 | 17 | 98 | 4 | 80 | 20 | 100 | 0 | 79 | 12 | 3.14 | 0.1342 |

RT, retention time; RRV, relative reduction value; SD, standard deviation; H, raw halloysite; HR, roasted halloysite; HA, activated halloysite; B, raw bentonite; BR, roasted bentonite; EV, expanded vermiculite; #, distinct odor (Lo et al., 2008); †, hazardous air pollutants (USEPA, 2011); §, 100% reduction efficiency signifies that a compound was not detected in the treatment group; $, Values followed by the same lowercase letter are not significantly different at $P < 0.05$ using the Tukey’s multiple-range test.
filtration in order to assess whether the method is limited to compounds that have a distinct odor. Therefore, future experiments should also involve olfactometry assessment or a combination of olfactometry with gas chromatography and mass spectrometry (GC-MS-O).

Moreover, the RRV of tentatively identified volatile compounds was obtained without estimating actual concentrations of compounds. However, it was assumed that the RRV calculated for estimates of concentrations would not be significantly different, as was explained and reported by Cai et al. [18].

4 Conclusions

The novel method of air purification inside livestock buildings, involving various types of aluminosilicates, was developed and tested for potential reduction efficiency with respect to ammonia and odorous compounds generated from poultry manure. It is worth noting that the examined device ODORI is suitable for commercially available sorbents and could be applied also for mitigation of odor emitted from other types of manure. The results of the present study showed that the effectiveness of the air scrubber packed with aluminosilicates is comparable to alternative methods that have been investigated in order to reduce volatile odorous compounds. Thus, considering the limitations of the present work, further studies are needed.

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