

Analysis of removing organic sulfur compounds from sewer off-gas by activated carbon

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Abstract

In the sewage system, there are a great number of biological/physical processes that release in the atmosphere odors which may be harmful for the environment and human health.

This master thesis is an attempt to gather more information about how a specific hybrid filter based on activated carbon behaves under definite laboratory conditions to prevent and eliminate the odors that are released in the atmosphere. Two laboratory experiments were conducted to establish the kinetics of the filter. To see the absorption capacity, 3 adsorption isotherm models were made: Linear, Langmuir and Freudlich for the chosen compounds: Methyl mercaptan (MeSH) and Dimethyl sulfide (DMS). These substances are part from volatile sulfur compounds group and are common in the sewage atmosphere. The adsorption capacity isotherms are made on filter impregnated with active carbon but also on filter support.

After conducting the experiments and comparing them to the theoretical data, it was concluded that the hybrid filter based on activated carbon acts properly even with a high concentration of volatile organic compounds.

Introduction

One of the most important infrastructure that human kind could possible invent is the sewage system which collects sewage and storm water altogether, depending on what kind of system chosen. But, with the advantage that this waste is not 'running' on the streets, also come some disadvantages. One that is an alarm signal is the odor emitted from the pipes. This odor is a gas which contains volatile sulfur compounds, nitrogen compounds and fatty acids. These compounds are a result from the biological and chemical reactions in aerobic/anaerobic conditions that occurs in the sewage system. Aerobic condition is when oxygen is present at the place of the experiment. Naturally, anaerobic condition is when there is no oxygen present.

Some of the reactions that include sulfur compounds are:

- sulfur reducing bacteria that reduce sulfate to hydrogen sulfide;
- amino acids decompositions that contains sulfur;
- methylation of mercaptans;
- oxidation of methyl mercaptan which generate dimethyl sulfide; (Il Choi, 2012)

These compounds are generated under anaerobic conditions, for example in pressure mains, but they are released at discharge manholes and at drop structure. (E. A. Rudelle, 2013)

The volatile sulfur compounds, including H₂S, not only are a problem regarding odor but also represent a problem regarding the corrosion of the pipes, which in time damages the efficiency of the pipes and, also, can result in leakages in the environment. This means that the leaked substances, solutions and odors could be harmful for the human health.

The dominating sulfur compounds that occurs in the sewer atmosphere are hydrogen sulfide (H₂S), methyl mercaptan (MeSH), ethyl mercaptan (EtSH), and dimethyl sulfide (DMS). (Eric C. Sivret, 2016)

Two of the dominating compounds were selected for this study for further determination. These compounds are methyl mercaptan and dimethyl sulfide.

Methyl mercaptan is an odor gas which is a product between methanol and hydrogen sulfide. It has the main purpose in the synthesis of other organic compounds like pesticides, fungicides, plastics, compounds from jet fuels. Even if it occurs like a natural product, it is an environmental and health risk. It causes different health problems when it is inhaled (dizziness, irritate the skin/organ which encounters, vomiting) and in a large dose can affect the central nervous system. (Gale, 2017)

The permissible exposure limit for methyl mercaptan set by OSHA (Occupational Safety and Health Administration) is 10 ppm (20mg/m³), while the recommended safety limit set by NIOSH (National Institute for Occupational Safety and Health) is 0.5 ppm and the limit for immediately dangerous to life or health is 150 ppm. (Mgmt, u.d.)

Dimethyl sulfide is a product resulted from the breakdown of dimethylsulfoniopropionate and from microbiological process from the methabolization of methyl mercaptan by bacteria. Dimethylsulfoniopropionate is an organosulfur compound found in marine phytoplankton. It also causes the bad odor of rotten seaweed. (Jaqueline Stefels, 2007). Dimethyl sulfide can cause environmental problems when dimethyl sulfoxide is naturally transformed by bacteria in DMS and disposed into the sewers. It is being used in organic synthesis, as a reducing agent in ozonolyses reaction, and as a presulfiding agent. (Jaqueline Stefels, 2007)

In case of DMS, the permissible exposure limit does not have a value set by OSHA. (United States Department of Labor , 2007)

Methyl mercaptan and dimethyl sulfide have an odor threshold of 0.0011 ppm and 0.0001 ppm, respectively, in wastewaters and sewage systems. The odor threshold is not dependent on the source of the odor. (Amirreza Talaiekhosania, 2016)

For this study, these two compounds were selected because of their natural abundance in sewage systems. Since there was limited information about the kinetics of the filter in relation with these compounds, it was a good opportunity to establish how the filter reacts and behave when in contact with methyl mercaptan and dimethyl sulfide in different experimental conditions.

An effective way to reduce or eliminate the compounds from sewer atmosphere is the use of activated carbon impregnated on varied materials.

Activated carbon is the final form of amorphous carbons after they were treated (heating, burning) under specific conditions. From this groups of carbons, carbon fiber, carbon powder and black carbon are the main ones. The activated carbon is provided by the charcoal which was subject of thermal treatment in absence of air. (Rheinehart, 2000) This modification of physical conditions it is of a significant importance for the finished product which, in order to be activated, has to have a relative large surface area and well developed submicroscopic network pores. The primary characteristics of an activated carbon are its physical proprieties (surface area, product density, mesh size, abrasion resistance, ash content) and its activity. The activity proprieties include pore size distribution which is the pore volume of a carbon and there are three categories: micropore (less than 100 Angstroms¹), mesopore (100-1000 Angstroms), macropore (larger the 1000 Angstroms). Due to these categories, the adsorption of different compounds in the gas phase on activated carbon can be anticipated. For the gas phase, the majority of the pores would be in the micropore region, and a broader pore size distribution for the liquid phase. But, nevertheless the pore distribution network should be wider in order to have a good adsorption of molecular sizes. (John Sherbondy, 2015)

¹One Angstrom – 10E-10 mm;

All the activation processes are done in order to enhance the adsorption process. Adsorption refers to the physical and chemical process in which the molecules adhere on the surface of the activated carbon. The physical process is due to London Dispersion forces (a type of Van der Waals forces), (IPEC, u.d.) and the chemical reaction or chemisorption is due to ionic, covalent or metallic bond that can appear at the active sites on the surface of the activated carbon (Christmann, 2012). The adsorption process depends on several factors like: pH of the used solution, pore size distribution of the active surface, temperature of the process, molecular size of the substances that adhere to the active surface and the efficiency that can increase or decrease.

The efficiency of the adsorption process increases when:

- Particle size of the impurities decreases;
- At low temperature;
- The solubility of the impurities decreases;
- The time contact is minimum;(Rheinehart, 2000).

Basically, the adsorption process can be described as the conceptual model presented in Fig.1 in which the adsorbent is represented by the material (in this case, activated carbon) that the adsorptive (odors) sticks to. Adsorbate is the substance, in gas phase, that is already adsorbed to the adsorbent. The dash lines represent the area where the process can occur and most of the adsorption and desorption process is taking place.

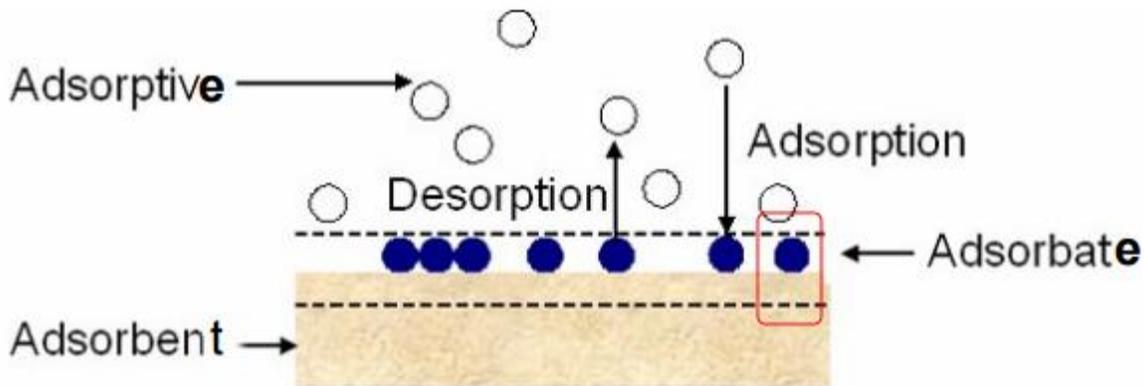


Fig.1 Conceptual model of adsorption process.(Christmann, 2012)

To see how a filter impregnated with activated carbon behaves under specific conditions, the following knowledge of adsorptions kinetics is essential. The information needed is the knowledge of how the adsorbate stays on the filter, which kind of adsorption (physical or chemical), the pore size distribution; these can be an indicator on how the filter behaves. In the case of this study, the activated carbon was granulated, which means that it is part of the mesopore, which means that it has a bigger importance when talking about gaseous compounds. To have a better understanding, some adsorption isotherm models were selected to predict the

maximum adsorb concentration of a volatile organic sulfur compound in a filter containing activated carbon.

Thesis objectives

To have a better understanding of the hybrid filter and to know what to look for in the experimental phase, the objectives of this thesis were:

- Increase the information about how the filter behaves under specific conditions;
- To determine an adsorption kinetic model;
- To determine an adsorption capacity in the case of the two organic volatile sulfur compounds;

2. Materials and methods.

To see the kinetics of the hybrid filter which is impregnated with activated carbon, laboratory experiments were conducted. The filter was provided by the Fritzmeier company and it is a hybrid activated carbon filter which his mainly purpose is retention of odor gases from sewage systems. In the below figure (Fig. 2), we can see the gas flow coming from the sewage system, as well as the water drainage that occurs inside the system.

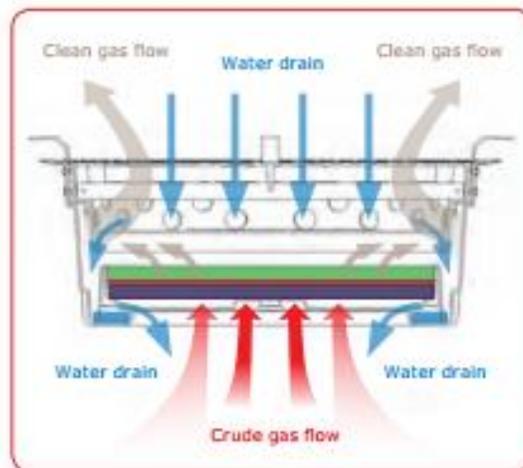


Fig. 2 Concept Model Hybrid Filter (Fritzmeier, 2015)

The filter is composed from 3 layers, one is impregnated with activated carbon (green layer), the second layer is a layer which is made from biomass that helps to eliminate ammonia compounds (red layer), and the third one is the filter support which is the same material that activated carbon was impregnated on (black layer). The filter was designed to treat naturally ventilated sewer air and developed to be incorporated into sewage manholes. (Fritzmeier, 2015)

To see how the filter reacts in diverse conditions two different experiments were conducted. In both sets of experiments, dimethyl sulfide (DMS) and methyl mercaptan (MeSH)

with a concentration of 10 ppm/100mL were used. 10 ppm is 10 parts per million which refers to the concentration in a liquid phase of the compounds in 100 milliliters of water.

2.1. Batch experiment

First type of experiment was conducted to see how the hybrid filter behave in an oxygen atmosphere (I), and a nitrogen atmosphere (II). 20 sets of experimental air samples were taken for every analyzed compound in both experimental condition. In this case, two hybrid filters with a diameter of 10 cm were used. Each of them were placed in a jar which was chain connected to a bottle of 500 mL placed on a magnetic stirrer which contains 10ppm/100 mL DMS/MeSH. The system was connected to a small Thomas G12/02 EB pump. The samples were taken from three places: one before the first jar, the second sample after the first jar which contains the filter, and the third sample after the second jar. The first sample was taken after 10 seconds after the pump was started, the second sample was taken after 1 minute from the first sample, and the third sample was taken after 1 minute from the second sample. It was decided to take the samples following this schedule because from the literature investigated, activated carbon has a very quick adsorption process. The second experimental condition (II) was conducted in the same way as the first one (I), the only difference is that before the experiment started the system was flushed 30 minutes with nitrogen. (Fig. 3)

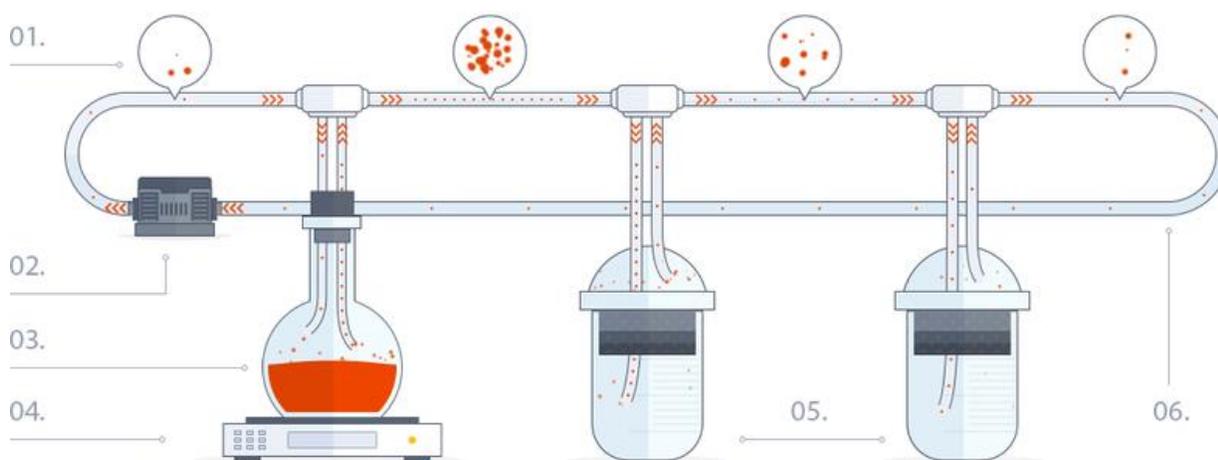


Fig.3 Schematic representation of batch experiment

The samples were taken with a 1 mL syringe in a 20-mL vial for analytical method which were flushed with nitrogen and hermetically sealed with Teflon coated serum cap. In Fig.3 there is a schematic representation of the actual set-up, where 1 is the point where the samples were taken, 2-pump, 3-the bottle with the substance (10 ppm DMS/MeSH), 4-magnetic stirrer, 5-jars with the activated filter; 6- connection between the jars.

2.2. Adsorption capacity experiment

Second type of experiment was conducted to see the capacity of different amounts of filter and filter support to adsorb volatile organic sulfur compounds, same compounds as in the

batch experiments (DMS/MeSH). The amounts of hybrid filter and filter support used for this experiment are 0,01g; 0,05g; 0,1g; 0,5g and 1g. Each amount was placed in a 100mL bottle and flushed with nitrogen before the experiments started. 5 sets of experimental air samples were taken. The experiment started when each bottle with the specific amount of filter/filter support was injected with 1 mL air gas taken from 200mL bottle solution with a concentration of 10 ppm for each compound (DMS/MeSH). After the compound was injected, the bottles were agitated manually couple of times and left 5 minutes before taking the samples for analytical determination. The samples were taken with an 1mL syringe in a 20 mL vial flushed with nitrogen and hermetically sealed with Teflon coated serum cap. The experimental set up is presented in the below figure. (Fig. 4)

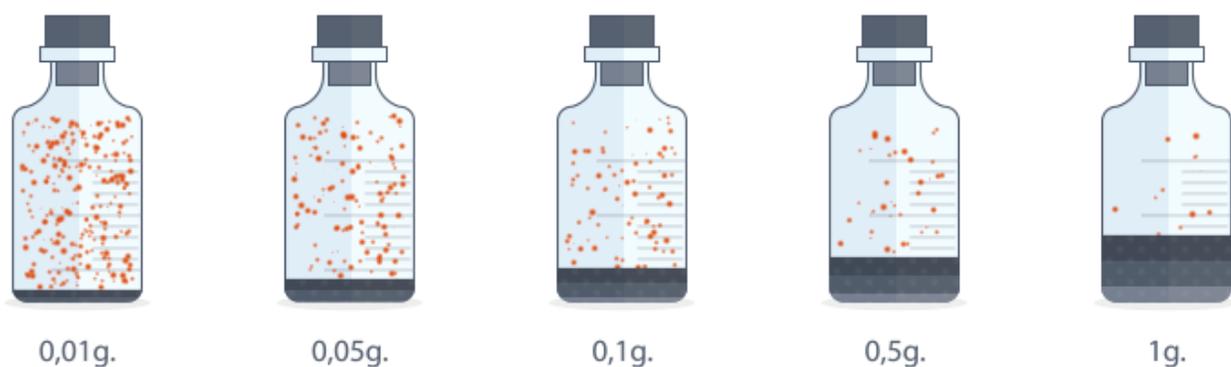


Fig. 4 Adsorption capacity schematics

2.3. Analytical method

To measure the analyzed compounds, DMS/MeSH, headspace chromatography with mass spectrometric detection (HS-GC-MS), from Thermo Fisher Scientific Inc., MA, USA (Trace GC Ultra + DSQ II) was used. The GC-MS has a column with L: 30m; ID: 0,25mm; film: 0,25 μ m; (TR-1MS) and the carrier gas was helium (1,3 mL/min⁻¹). The volumes samples that was injected in the detector was 0,5 mL at 220°C in split mode. The measured method was ion-monitoring mode and both compounds were calibrated using external standards with known retention time which are shown in Table 1.

Compound	Analytical ion (amu)	Retention time (min)	Material for experimental compounds
MeSH	48	1,48	Sodium methanethiol CH ₃ SNa 21% in H ₂ O
DMS	62	1.79	Dimethyl sulfide C ₂ H ₆ S

Table 1. Data quantification for GC-MS.(E. A. Rudelle, 2013)

3. Results and Discussion

3.1. Batch experiment

After the experiments were performed and passed through GC-MS, the chromatograms were analyzed and compared using the area of the peak of the compound that was passing through the experimental set-up. Due to the fact that it was a chained experiment, the peak which appears at time 1.79 minutes, usually has a wider area than the following ones (Chromatogram 1 from Fig. 5). After passing through the filters, the areas are very small (in case of manually selection) or not at all (in case of automatic detection of the peak using the software of the GC-MS). In the second and third chromatogram from Fig. 5 can be seen that at the time 1.79 minutes, there is no identifiable substance. Figures 6 has the same tendency in regards with the DMS peaks.

In Figure 7, the retention time for MeSH is at time 1.48 minutes and follows the same tendency as in the DMS case. For Figure 8, which has a nitrogen atmosphere, there can be seen a second peak at 1.79 minutes, which could mean that MeSH can be oxidized to DMS. But in the last chromatogram there are no peaks for either MeSH and the appeared peak at 1.79 minutes.

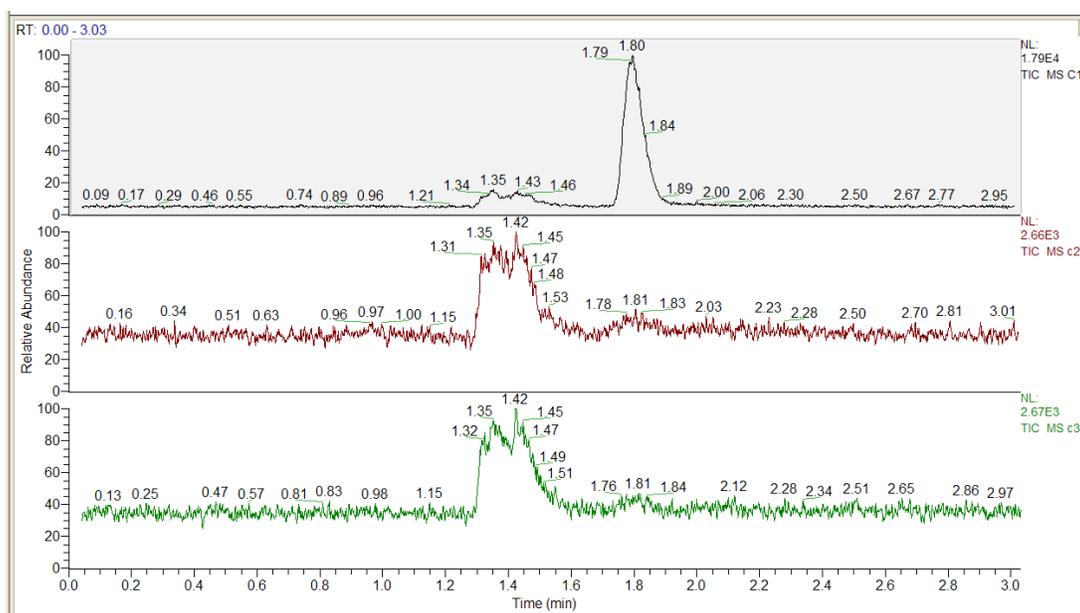


Fig.5 DMS chromatogram in air

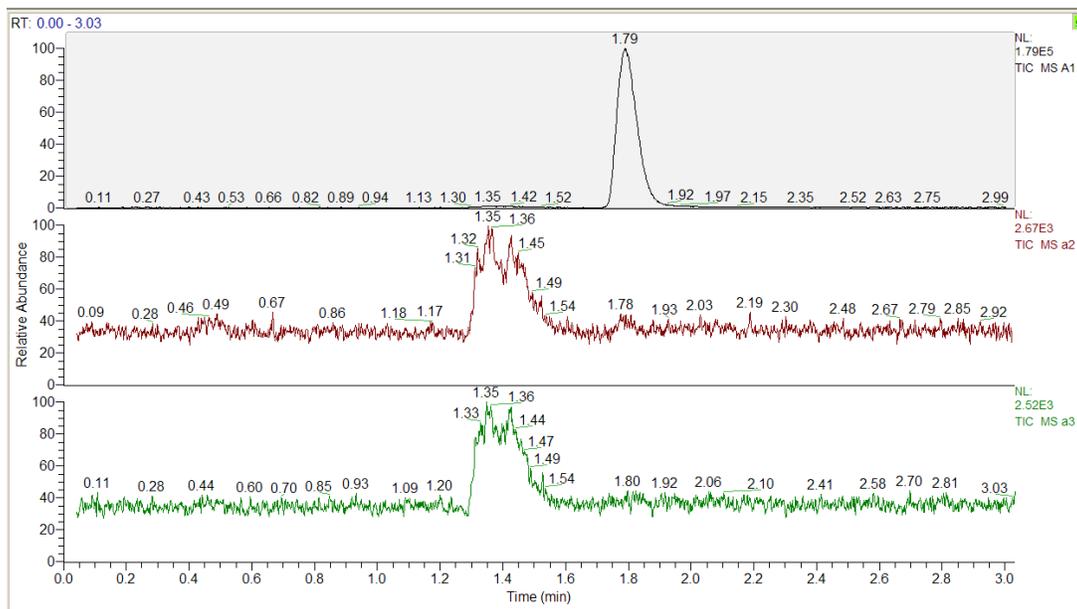


Fig.6 DMS chromatogram in N2

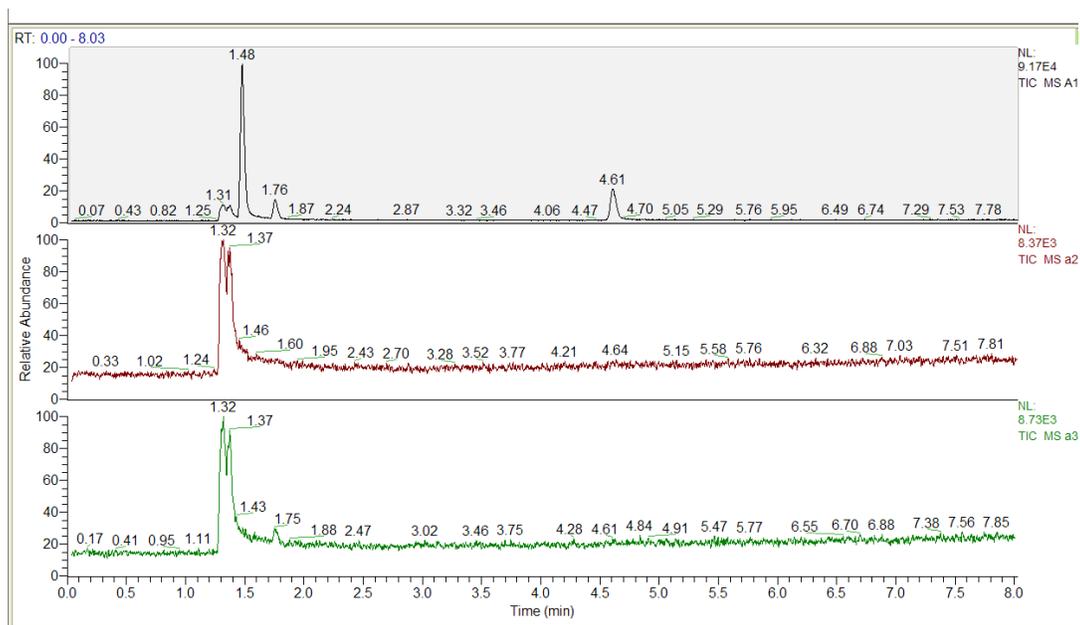


Fig.7 MeSH Chromatogram in air

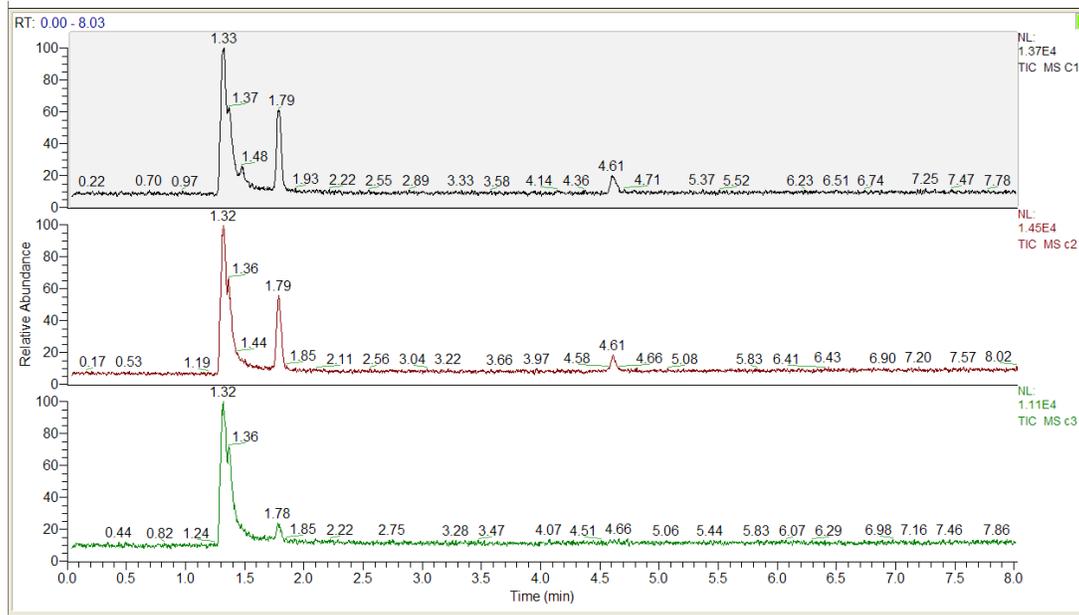


Fig.8MeSH chromatogram in N2

3.2. Adsorption capacity experiment

To predict an adsorption capacity, 3 isotherm models were made: Linear, Langmuir and Freundlich for filter and filter support. These models are calculated for linear and nonlinear regression using Excel Solver tool.

Equation 1. is the linear function where ‘q’ is the adsorption capacity of the adsorbent, ‘c’ is the concentration and ‘m’ and ‘b’ are parameters that are adjusted with Solver tool to get a better fit.

$$q = mc+b \quad (\text{Eq.1.})$$

The Langmuir model is using the same Solver tool for getting a better fit of the two fitting parameters, Eq.2., where ‘q’ is the adsorption capacity of the adsorbent, ‘c’ is the concentration and ‘q_{max}’ and ‘K_l’ are the fitting parameters that are adjusted with Solver tool to get an optimal fit.

$$q = q_{\max} \frac{KlC}{1+KlC} \quad (\text{Eq.2.})$$

The Freundlich functions, Eq.3., also uses Solver tool for a better linearization of the plots, where ‘q’ is the adsorption capacity of the adsorbent, ‘c’ is the concentration of the substance and ‘K_f’ and ‘c^{1/n}’ are parameters that are calculated with Solver tool for a better fitting.(Dentel, 2016)

$$q = K_f C^{1/n} \quad (\text{Eq.3.})$$

In order to see the model that fits the best, ' r^2 ' has been calculated which is $1 - \text{RSS}/\text{TSS}$, ' RSS ' is the residual sum of squares and ' TSS ' is the total sum of squares. To verify this number, it has to match with the ' R square' given by Excel build-in linear regression. The model with the higher ' r^2 ' is the model with the best fit. (Dentel, 2016)

To have a better understanding of the term sum of squares, we can think of it as the sum of the squared variables of the linear regression. The residual sum of squares is the unpredicted part of the result. That means that if, for example, we have a point above the regression line, the distance from that point to the regression line is the residual, because we cannot explain what influenced the point to be above the line.

In Fig.9 the models are calculated for DMS in case of the filter. The best model fit with the higher ' r^2 ' is Langmuir model with a value of 1,000, while the Linear has a value of 0,908 and Freundlich 0,886. As we can see, the linear and Langmuir models are almost identical.

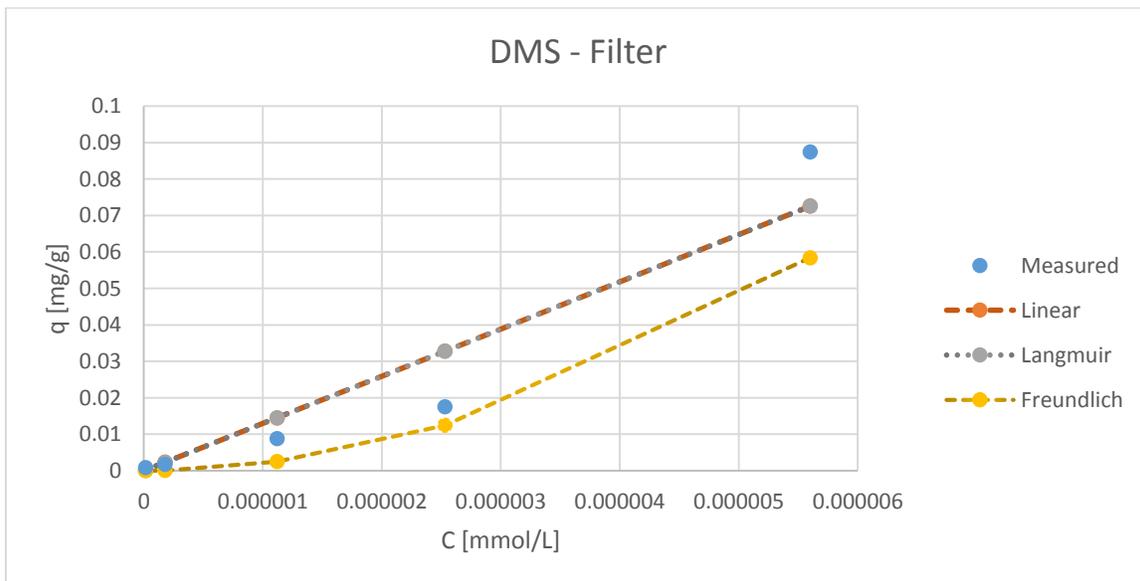


Fig. 9. Adsorption isotherm models for DMS – Filter

In Fig.10 are presented the models in case of DMS with the filter suport. In this case the values of ' r^2 ' for Linear and Langmuir adsorbtion isotherm model are equal, 0,998, and Freundlich has a value of 0,487.

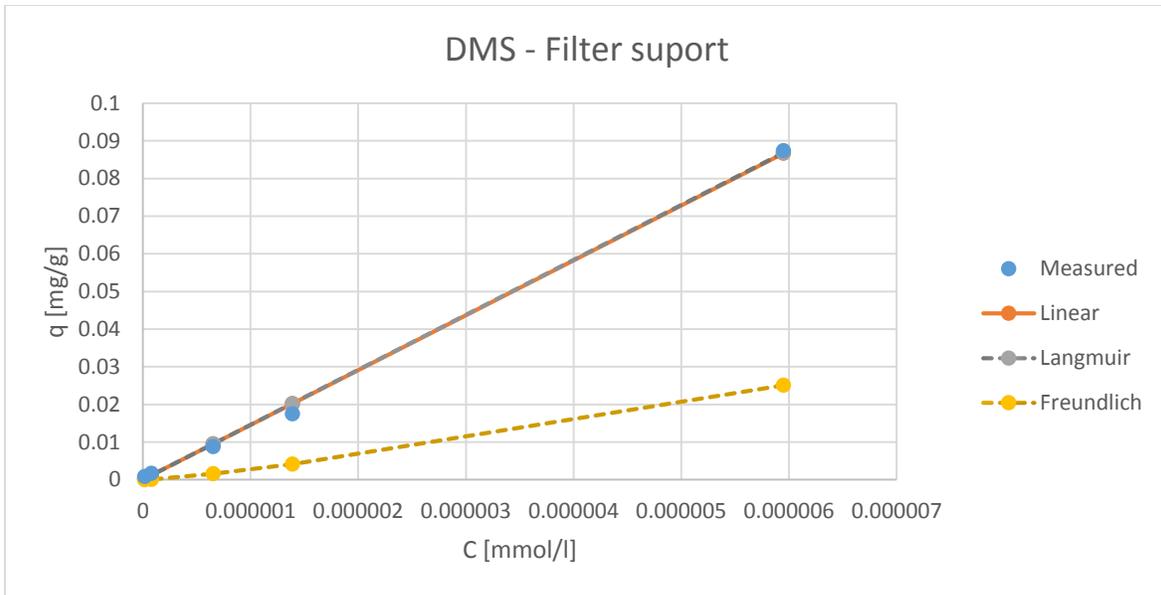


Fig.10 Adsorbtion isotherm models for DMS – Filter suport

In both cases from above, ‘ r^2 ’ matches the solution offered by the Excel Data Analyzer which is very close to the calculated ones.

For the second compound, MeSHwas introduced in the same volume with different amounts of filter/filter support.

In Fig.11 it is presented the models for the adsorption isotherm models, in this case ‘ r^2 ’ for Langmuir and Linear are 0,122, both with the same value, and Freundlich is 0,85. It can be said that Freundlich model is a suitable model even if ‘ r^2 ’ calculated by Excel Data Analyzer has value close to 1,000.

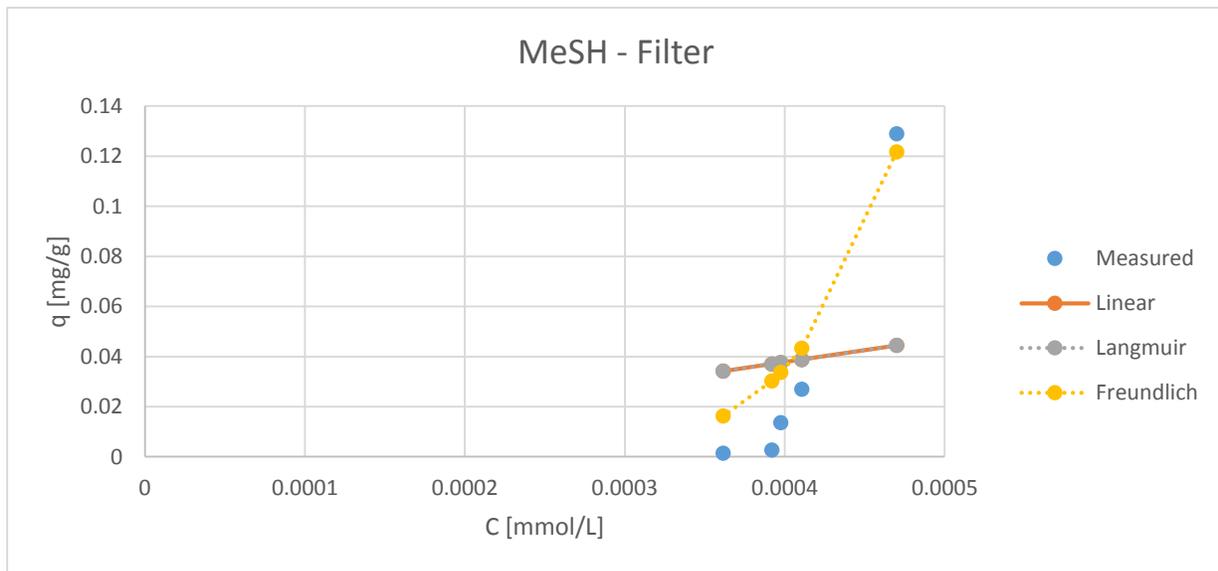


Fig. 11 Adsorption isotherm moedels for MeSH – Filter

In Fig.12 are presented the models for MeSH in case of filter suport, where, none of the calculated models are close to the measured ones. The values of 'r²' are closer to zero, 0,140 for Linear model, 0,416 for Langmuir model, and in the case of Freundlich model the value is - 0,510.

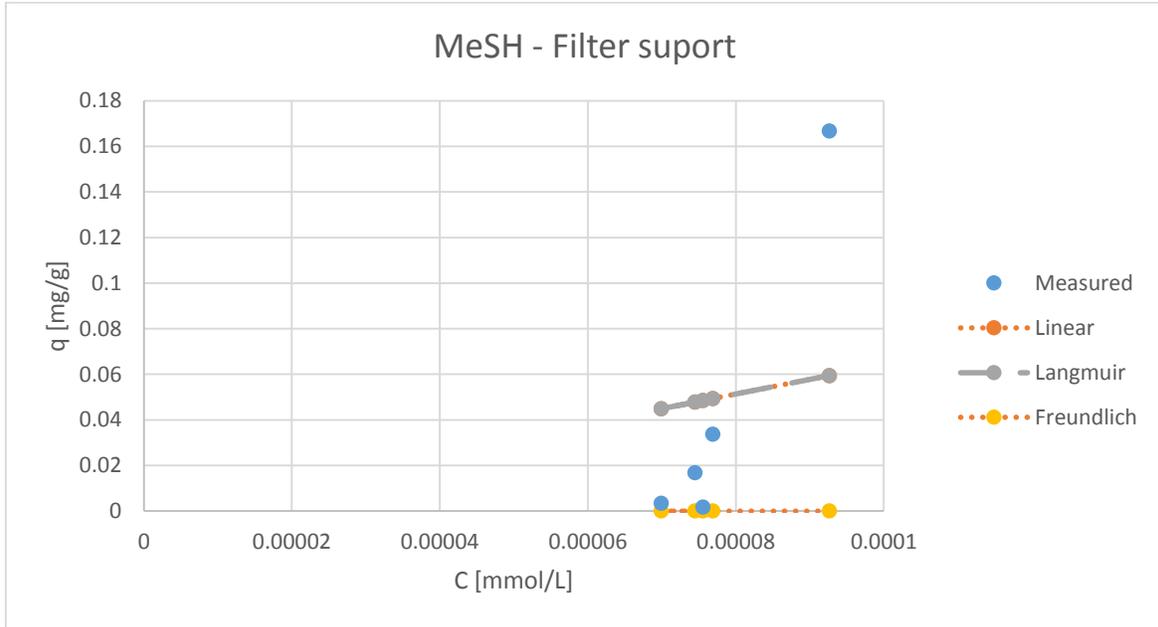


Fig.12. Adsorption isotherm models for MeSH – Filter suport

From the above adsorption isotherm models, we can observe that in the case of DMS, the made models followed the experimental data, but in the case of methyl mercaptan, the experimental data does not follow the models. A explanation for this could be the nature of the compounds, because it can be oxidized very quicly, which means that it can be difficult to manage.

From a theoretical point of view, the data set is not sufficient enough as to reach the point of saturation. This means that there would be a need for at least 10 more samples to have a complete model. In the figure below (Fig. 13) we can see all three models, and how they should be represented theoretically.

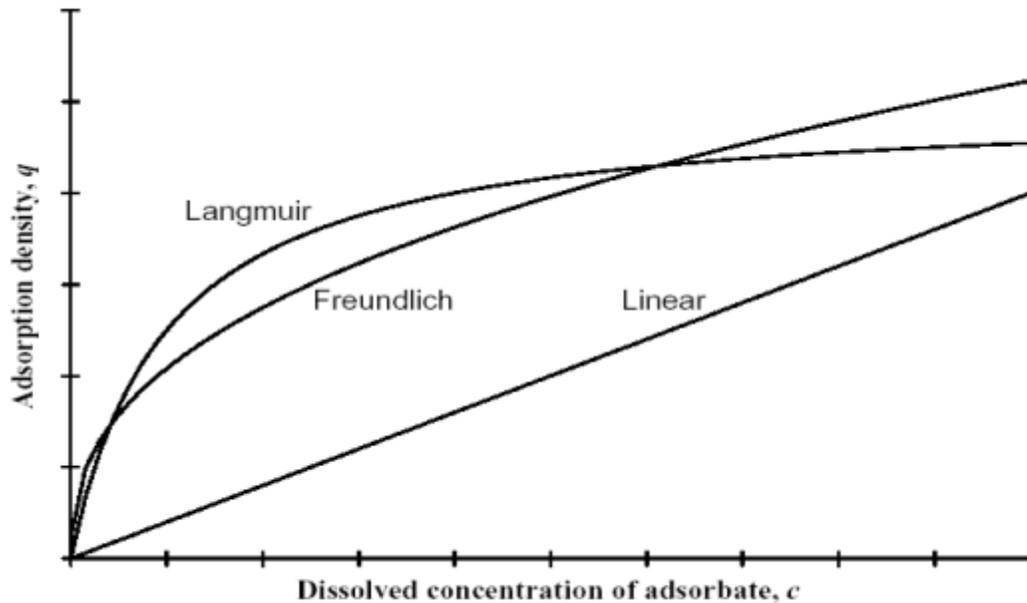


Fig. 13 Theoretical view of the three isotherm models (Faculty of Washington, u.d.)

From all of the above, it can be seen that only in the case of the DMS adsorption isotherm model there is a resemblance with the theoretical model. This is limited to the beginning of the slope, before reaching saturation points.

The filter impregnated with active carbon behaves like it was expected, adsorbing most of the compounds even in small quantities.

The filter support behaved almost like it was impregnated with active carbon, this can be due to contacting the filter when it was transported, handling, and the activated carbon stuck to the material support. Or, another reason that the filter support behaved like this, could be from the manufacturer. It could be treated with different substances for a better adsorption of sulfur compounds or other ones.

After the process of adsorption begins, the compound methyl mercaptan is usually found in the micropores of the filter and/or it is oxidized to produce dimethyl disulfide, which is strongly adsorbed. The DMS compound, because of its similar molecular weight with dimethyl disulfide, share the same adsorption rate. (Ari Shammaya, 2016)

A study that was conducted in Australian sewage systems regarding odors and the removal of them using different technologies and materials impregnated on filters concluded that activated carbon has a high chemical/physical stability, a large surface area (400-1200 m²/g) and a high porosity in comparison with other materials like peat, coconut shell, compost, sewage sludge, porous ceramic, plastic packing and others. Also the lifespan of the activated carbon is longer than 5 years in comparison with the other beds suitable for odor removal, which, except soil media (10-20 years), have a lifespan that is up to 4 years or less. (Ari Shammaya, 2016)

In terms of removing volatile sulfur compounds, the percentage is up to 100%; in case of methyl mercaptan has a good consistency between 77-100% removal rate, and for dimethyl sulfide the consistency is poor but the removal rate is between 75-93%. (Ari Shammaya, 2016)

The adsorption capacity experiments follow the removal rate from the Australian study. Even at the smallest amount of filter impregnated with activated carbon, the adsorption of the compound was high. Both experiments, in case of DMS and MeSH, have the same tendency of removal rate.

The removal rate of the compounds depends on the contact time with the filter. Using a EBCT (empty-bed contact time) values, it can be assumed that the optimal contact time should be 60 seconds. The first 20 seconds being used in adsorption of hydrogen sulphite, while the rest of 40 seconds would be used on adsorption of volatile organic compounds, including DMS and MeSH.(Joyce, 2015)

In a literature review, for an optimal adsorption, the superficial gas velocity should be between 500-1800 m³/m²h.(Ari Shammaya, 2016)

Using the same data, in Fig.14 and Fig.15, there is presented the adsorption kinetics of the adsorbent at a specific time, which is calculated using Eq.4. where 'qt' is the adsorption capacity of the adsorbent at time 't', 'C₀' and 'C_t' are the initial and final concentration, 'V' is the solution volume, and 'm' is the mass of adsorbent.

$$q_t = (C_0 - C_t) * V / m \quad (\text{Eq.4.})$$

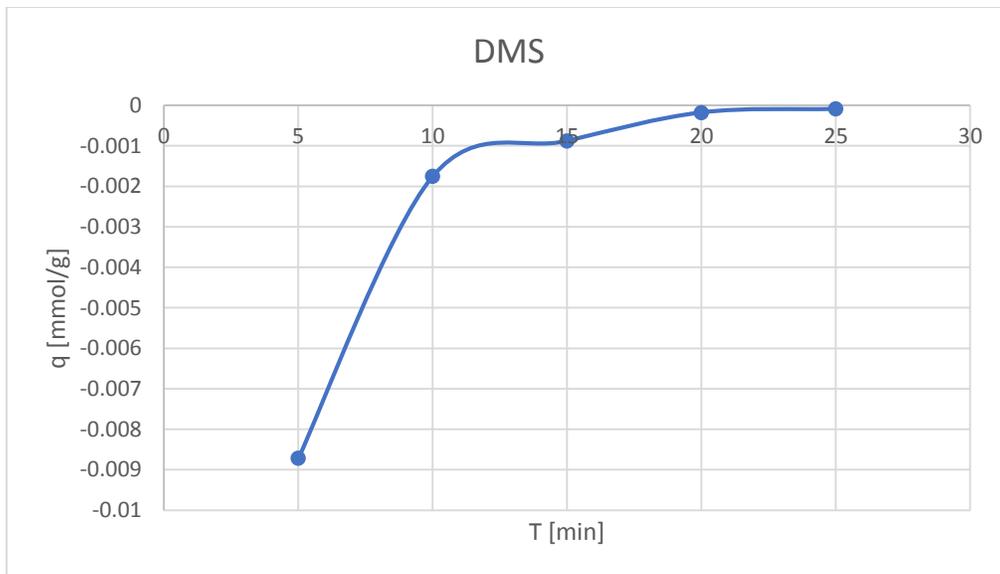


Fig.14 Adsorption kinetics at the specific time for DMS

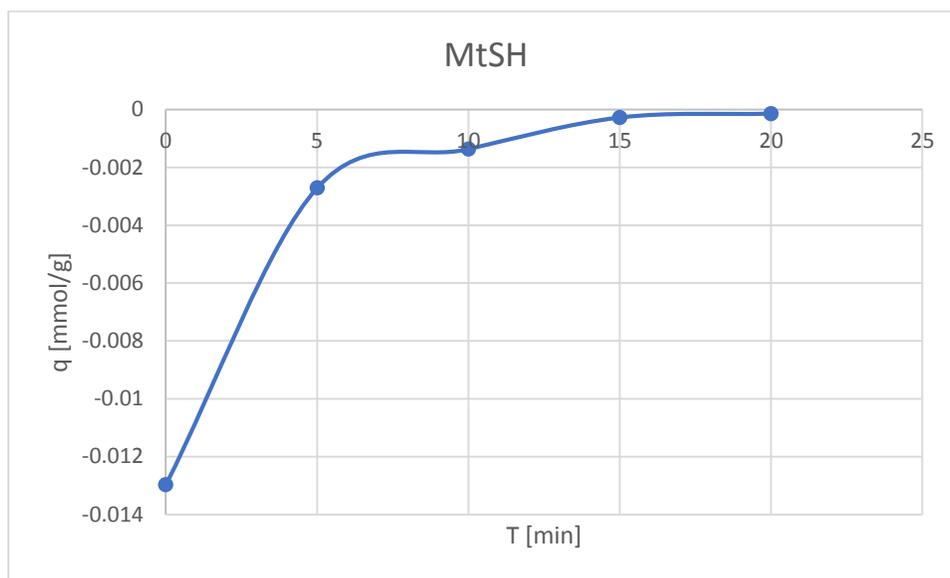


Fig.15 Adsorption kinetics at the specific time for MeSH

It can be observed that, in both cases, the adsorbed kinetics over time is increasing after the first 10 minutes, with an adsorption rate of up to 70% of the concentration. After that, it reaches a constant removal rate of approximately 100%.

4. Conclusions

From the first type of experiments, batch experiments, can be concluded that the kinetics of the filter are fast. Even when the concentration of the substances that are going through the system are relatively high in comparison with the threshold values, the adsorption is taking place immediately. The contact time with the filter is approximately 1 minute after the pump was started, and the GC-MS chromatograms showed that there is no substance after the first jar, and if there was some, it is even less or close to zero, after the second one. The same results are in the case of the nitrogen atmosphere.

From the second type of the experiments, there can be concluded that it is close to reach an equilibrium constant, but because of the lack of data points this is not accomplished in the DMS case for the filter and filter support.

In the MeSH case, a conclusion is difficult to draw because the adsorption isotherm models are not similar to the theoretical model, even though the adsorption process is taking place; it is not reaching an equilibrium.

4.1. Practice

In practice, the information found after the experiments were performed will be helpful in order for a better understanding of how the filter will behave when a high amount of odors will pass through it and how effective it will be. Even at high dosage the adsorption is taking place in the first minutes, with or without oxygen being present, and it is known, from the literature review, that the lifespan of the filter based on activated carbon is more than 5 years. (Ari Shammaya, 2016)

To have an approximation of the lifespan, several conditions to delimitate can be used. With conditions like a contact time of the compounds to the filter to 60 seconds, an inlet flow velocity between 500 and 1800 m³/m²h, with a concentration equal to the threshold odor, meteorological conditions of the northern hemisphere, proximity to contamination source and other. However, the removal rate of the filter depends on air flow, if the flow velocity is between the limits specified above, the contact time will be sufficient that the compounds are completely eliminated. But, in case the air flow is above the maximum limit the contact time will not be enough so that the compounds can be adsorbed.

Using the above conditions after gathering of more data points for the adsorption isotherm models, a lifespan can be estimated.

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