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Macro-Kinetics of Biofiltration for Odor Control: Dimethyl Disulfide

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Abstract

A dual-column biofilter system with two different composts was used to investigate the macro-kinetics of dimethyl disulfide (DMDS) degradation. The biofilter columns were filled with compost mixtures up to one meter. The gas flow rate and DMDS concentration to the biofilters were varied to study their effect on the removal characteristics of DMDS. It was found that the biodegradation of DMDS was governed by zero-order reaction-limited macro-kinetics for inlet DMDS concentrations between 10 and 55 ppmv. The overall average zero-order kinetic coefficient for DMDS removal by compost was 0.50 (± 0.1) ppm/sec for both compost mixtures studied. Variations in individual kinetic coefficients were observed due to varying environmental conditions, such as pH and temperature. The kinetic coefficients determined are specific to the system discussed in this work. During high acidity conditions in the filter beds, methyl mercaptan (MM) was observed in the gas samples collected. Appearance of MM was probably due to decreased microbial activity in the lower portions of the biofilter. Considering the neutral pH range required and the presence of methyl mercaptan, it is likely that the microorganisms present in the biofilters used in this research are similar to the T. thioparus (strain E6) species.

Key words : biofilter, kinetic model, odor, methyl mercaptan, sulfur compound

1. INTRODUCTION

Odorous gas emissions from commercial and industrial activities are frequent targets of public complaint. Organo-sulfur compounds commonly exist in malodorous emissions from industrial sources. Important odorous sulfur compounds are carbonyl sulfide (COS), carbon disulfide (CS_2), lower molecular weight mercaptains (RSH), sulfides (R_2S) and thiophenes (C_4H_4S). Major sources of these reduced sulfur compounds (RSCs) are paper and pulp industries, waste water treatment plants, chemical manufactoring industries and petroleum refineries (Ruokojarvi *et al.*, 2001; Smet *et al.*, 1998). Hydrogen sulfide (H_2S) which is usually emitted in comparatively large amounts and tends to mask the odor of the organo-sulfur compounds.

The majority of biofilter research and development activities has been performed in Europe and Asia, particularly in Germany and Netherlands. The introduction of biofilter applications in the U.S. and Korea has been slow compared to the European countries, probably due to the limited avalilability of information on the fundamental kinetics and mechanisms of pollutant removal.

Mathematical models describing the biofiltration process are of utmost importance in the design of indu-

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strial-scale biofilters. Data gathered from laboratory-scale studies can be used in a mathematical model to obtain necessary design parameters for pilot- and full-scale systems. There are two main types of kinetic studies in biofiltration, namely, micro-kinetics and macro-kinetics. The micro-kinetic analyses aim at describing the individual micro-processes taking place in a biofilter such as microbial degradation, whereas macro-kinetic analyses involve determination of the overall effect of all the processes occurring such as mass transfer, adsorption, etc.

There are several studies that have been conducted on the macro-kinetics of biofiltration removal of pollutants. Shareefdeen *et al.* (1993) developed a numerical model describing biofiltration of methanol vapor. The model used double-substrate limited growth, where dependence on methanol was described by Andrews-type kinetics and dependence on oxygen was described by Monod-type kinetics.

Hodge and Devinny (1995) proposed another model that described transfer between the air and solid/water phases, biological degradation of substrate, CO_2 production and accumulation, and pH changes resulting from CO_2 accumulation. The model employed first-order Monod-type microbial degradation kinetics. The latter model equations were solved using two-step, explicit, finite difference approximation techniques.

Deshusses *et al.* (1995a, b) described a novel diffusion reaction model for the determination of both steady-state and transient-state behavior of biofilters. They used methyl ethyl ketone and methyl isobutyl ketone vapors to illustrate and discuss the model's results. Simultaneously using two different pollutants has the advantage of taking into account pollutant interactions.

All of the models mentioned previously involve, however, complex mathematical solutions which require the use of numerical methods. Simpler, yet effective models involving analytical solutions may have more practical applications. A model developed by Ottengraf and his co-workers is just such a tool (Ottengraf, 1986; Ottengraf and Van den Oever, 1983; Ottengraf, 1977). Ottengraf group's model has been widely accepted and used in macro-kinetic studies of biofiltration (Phatak, 1993; Yang, 1992).

An organo-sulfur compound studied in this research work is dimethyl disulfide (DMDS), and the model was also used in the present study on DMDS biofiltration. The objective of this study is to analyze the macro-kinetics of degradation of DMDS in a compost biofilter, specifically, to obtain quantitative data on the kinetic order of reaction and reaction rate coefficients. The macro-kinetic model developed by Ottengraf (1986) is applied to the analysis of DMDS concentration decay in gas samples collected along the biofilter columns. In addition, the potential bacterial strain involved in this biofilter work will be discussed based on this and other microbiological studies.

2. EXPERIMENATAL METHODS

2. 1 Experimental setup and operations

A laboratory-scale dual-column compost biofilter system used in this study was developed by the Air Pollution Group at the University of Florida and modified to investigate biodegradation of DMDS in an air stream. Originally, the biofilter system was designed, constructed and used in the biofiltration control studies of hydrogen sulfide by Yang and Allen (1994).

The biofilter system consisted of an air blower, a humidification chamber, a syringe pump, and two identical biofilter columns that were operated in parallel. A half horsepower blower was used to move room air containing pollutant vapor through the biofilter system. Before introduction to the biofilter columns, the room air was first treated in the humidification chamber. The chamber housed a spray nozzle which atomized tap water. In order to increase the amount of contact between the counter-current air flow and the water streams, the humidification chamber was filled with 5 cm diameter pall rings almost up to the nozzle level. The room air leaved the humidification chamber with greater than 95% relative humidity. Humidified air was transported to the biofilter columns via PVC pipes.

Before entering the biofilter columns, liquid DMDS (CAS#624-92-0, 99% purity, ACROS Chemicals, Pittsburgh, PA) was injected into the humid air flow using a syringe pump (Model 355, Sage Instrument, White Plains, NY). The biofilter columns were made of transparent cylindrical Acrylic tubes, 0.15 m inner diameter and 1.34, length. At the top and bottom of the columns access ports for the introduction and drainage of liquids were provided. Contaminated air was fed into the compost biofilters at the bottom of the columns. Several ports were located along the vertical packed columns to allow for measurement of gas and compost properties. The columns were packed with compost up to a height of one meter. A perforated Acrylic plate locater above the column inlet was used to support the compost and uniformly distribute the incoming polluted air.

2. 2 Selection of the filter materials

As a preliminary study, seven different filter materials (compost mixtures) were tested in order to determine the best two materials for treating dimethyl disulfide (DMDS). The filter materials were obtained from Atlas Peat and Soil, Inc., Boynton Beach, FL, USA. The study was conducted in a batch mode, where the filter material samples were placed in one liter flasks and equal amounts of DMDS gas were supplied to each sample. The gas residence time in each flask based on empty bed was adjusted to 1.5 minutes. The tests were done in duplicates. The best two filter materials determined from the preliminary study were used in this biofiltration studies of DMDS.

The filter material packed in the first biofilter column (#1) was a compost/pine mulch mixture whereas the material packed in the second column (#2) was a compost/bark nugget mixture. Due to the rather large size of the bark nuggets, the second column biofilter material had a higher percentage of large particles. In fact, more than 50% of the particles in the second column were larger than 9.5 mm (equivalent diameter). The larger particle sizes used in the second column may have had an advantage in terms of pressure drop across the column but were at a disadvantage in terms of providing an adequate active surface area for microbial growth and retention.

Compost mixture were used in the biofilters without any preliminary size or microbial treatment. The filter materials may be sieved, however, to remove small particles, in order to decrease pressure drop. The filter materials are generally seeded with activated sludge from wastewater treatment plants to introduce the microorganisms necessary for removing the pollutants poorly biodegradable (Ottengraf *et al.*, 1986). A disadvantage of using activated sludge is that it can clog the biofilter and create excessive pressure drops. It is also known that composts are rich in the number and variety of microorganisms are necessary for biodegradation. However, the compost mixtures used in this study were not seeded.

Few studies have been conducted on the microbiology of DMDS degradation. Early studies, such as those by Kanagawa and Kelly (1986) using Thiobacillus thioparus and by De Bont *et al.* (1981) using Hyphomicrobium, showed that these microbial species can consume DMS but can not utilize DMDS. Suylen and Kuenen (1986) reported a mixed culture enriched on dimethyl sulfoxide (DMSO) that could oxidize DMDS at 8% the rate of DMS oxidation. These facts suggest that the ability to grow on DMDS was not a property necessarily common to DMS-using bacteria and indicated that specific enrichment using DMDS as a substrate was necessary to produce DMDS-oxidizing bacterial strains (Smith and Kelly, 1988a). A list of microorganisms known to degrade DMDS is displayed in

Table 1. Summary of microorganisms known to degrade DMDS.

Microorganism name	Investigators
Thiobacillus thioparus, strain E6	Smith and Kelly (1988a, b)
Thiobacillus thioparus, strain DW44	Shoda (1993); Cho *et al.* (1991c, 1992a, b); Park *et al.* (1993a, b)
Thiobacillus thioparus, strain TK-m	Kanagawa and Mikami (1989)
Hyphomicrobium sp. I55	Zhang *et al.* (1991)
Basidiomycete sp.	Phae and Shoda (1991)
Cephalosporium sp.	Ishikawa *et al.* (1980)

Table 1.

2. 3 Methods of sampling and analysis

Following system dynamic equilibration, gas samples from the ports were slowly expanded into Tedlar bags using the slight positive pressure of the carrier gas in the biofilter columns. Gas samples in the Tedlar bags were analyzed, within the same day, for sulfurous compounds using a Gas Chromatograph/Flame Photometric Detector (GC/FPD) (Model 250H, TRACOR Inc, Austin, TX). The detector signals from the GC/FPD were processed using an electronic integrator (Model SP427, Spectra Physics, San Jose, CA). The GC separation was carried out using a 30″ × 1/8″ Teflon column packed with Super Q 80/100 (Alltech Associates, Inc., Deerfield, IL). The limit of detection for the analytical method was estimated to be 1.0 ppmv DMDS. Therefore, the concentration range studied in this study was limited to values above 10 ppmv.

2. 4 Biophysical models for a biofiltration of DMDS

Ottengraf's biophysical model is represented in Figure 1. According to this model a wet biolayer of thickness, δ, surrounds the filter particle. The following assumptions are made in the model (Ottengraf, 1986):

- The interface resistance to mass transfer in the gas phase can be neglected and the biolayer pollutant concentration at the interface may be assumed to be in equilibrium with the pollutant concentration in the bulk gas phase;
- In the biolayer, pollutants (substrates) are transported by diffusion, which is described by an effective diffusion coefficient D';
- The biolayer thickness, δ, is small compared to the diameter of the filter material particles;
- The micro-kinetics of substrate elimination reactions in the biolayer are described by Monod kinetics; and
- Flow of gas through the filter bed is plug flow.

Using these assumptions, the following differential equation describing the pollutant concentration, C, inside the biolayer can be written:

$$D \cdot \frac{d^2C}{dx^2} - R = 0 \tag{1}$$

where, x is the distance along the biolayer and R is the microbial degradation rate.

The micro-kinetics of microbial degradation is described by the following Monod equation:

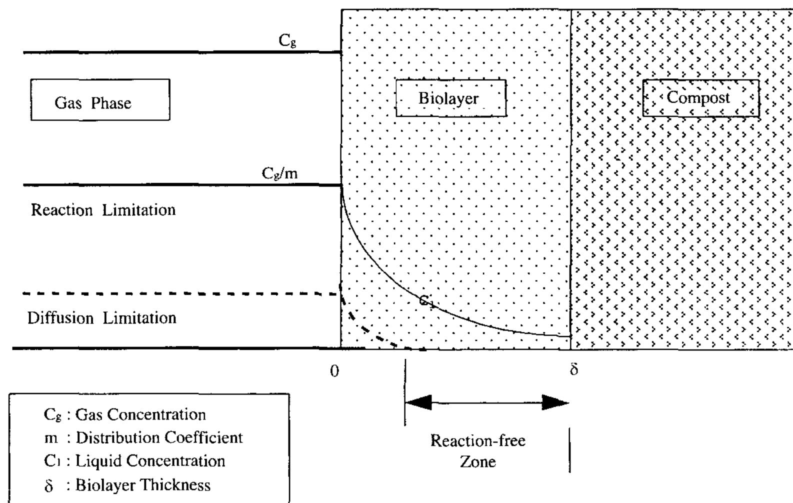


Fig. 1. Graphical representation of a biophysical model for a biofilter (Source: Ottengraf and Van den Oever, 1983).

$$-\frac{dC}{dt}=\frac{V_{max}\cdot C\cdot B}{(K_s+C)} \quad (2)$$

where V_{max} is the maximum reaction rate, K_s is the half-saturation constant, B is the microbial population density and t is the reaction time.

The Monod kinetics equation can be simplified for certain situations. When $K_s \gg C$, first-order kinetics result and the equation is reduced to:

$$\ln\left(\frac{C_0}{C}\right)=k_1\cdot t \quad (3)$$

where k_1 ($=V_{max}\cdot B/K_s$) is the first-order micro-kinetic coefficient, C_0 is the initial pollutant concentration.

When $k_1\cdot C$ is substituted for R in Equation (1), a macro-kinetic equation similar to the micro-kinetic equation (3) can be obtained:

$$\ln\left(\frac{C_0}{C}\right)=K_1\cdot t \quad (4)$$

where K_1 is the first-order macro-kinetic coefficient.

When $C \gg K_s$, the microbial reaction rate is said to be zero-order and the Monod equation reduces to:

$$C_0-C=k_0\cdot t \quad (5)$$

where k_0 ($=V_{max}\cdot B$) is the zero-order micro-kinetic coefficient. Substituting k_0 for R in Equation (1) and solving the differential equation yields the corresponding macro-kinetic equation. However, for zero-order microbial degradation Ottengraf and Van den Oever (1983) describe two situations. Referring to the biophysical model depicted in Figure 1, these conditions are:

- Reaction limitation: There is no limitation in the diffusion of pollutants (substrates) into the wet biolayer from the gas phase. Degradation of the pollutant is only controlled by the microbial degradation rate. In this situation the biolayer is fully active (solid line in Figure 1).
- Diffusion limitation: Diffusion of pollutants into the wet biolayer is limited. Thus, the pollutants can not penetrate through the whole biolayer. Therefore, the biolayer is not fully active and there are reaction-free zones in the biolayer. In this case diffusion is the rate limiting step (dashed lines in Figure 1).

For reaction limited conditions the macro-kinetic equation can be written in a manner similar to zero-order micro-kinetic equation (5) as:

$$C_0-C=K_0\cdot t \quad (6)$$

where K_0 is the zero-order macro-kinetic coefficient.

The macro-kinetics for diffusion limited degradation provides for a fractional order dependence that is developed in a simple form:

$$1-\sqrt{\frac{C}{C_0}}=K_f\cdot t \quad (7)$$

where K_f is the fractional-order macro-kinetic coefficient.

3. RESULTS AND DISCUSSION

3.1 Modeling of biofiltration

The locations of the gas sampling ports were matched with empty bed residence times to determine the degradation rate of DMDS. Note that the pollutant residence time in this study is determined as the "empty-bed residence time". In general, physical parameters of the filter materials such as porosity, structure, etc. affect the residence time of gas. A typical DMDS concentration profile along the biofilter column is shown in Figure 2. As can be seen from this figure, consumption of DMDS follows a linear trend with residence time.

In order to determine the kinetic dependence of DMDS loss, plots of (C_0-C), ln (C_0/C) and $1-(C/C_0)^{0.5}$ versus residence time were prepared to represent zero-, first- and fractional-order kinetics, respectively. Linear least squares regression analyses were applied to each plot to determine the kinetics with the best fit (highest correlation coefficient). For all the kinetic concentration data collected, zero-order plots showed the highest correlation coefficients, suggesting that the

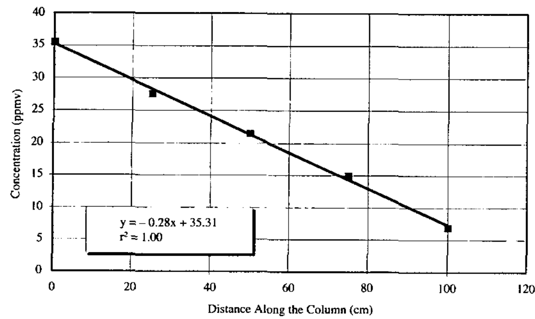


Fig. 2. Typical DMDS concentration profile along the biofilter (Flow Rate = 18.3 Lpm).

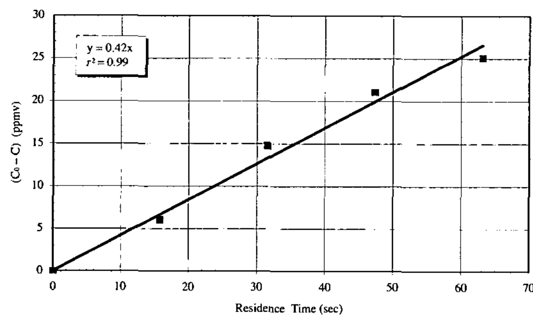


Fig. 3. Plot showing Zero-Order kinetics of DMDS removal for the first column compost mixture (Initial Conc. = 25.1 ppmv, Gas Flow Rate = 16.8 Lpm).

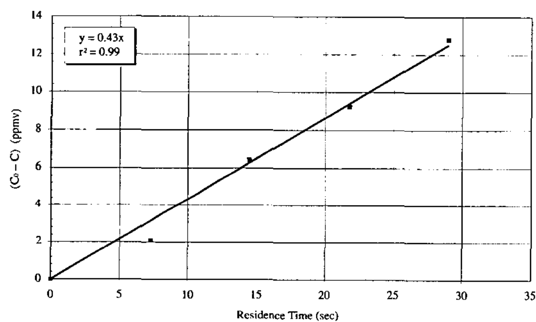


Fig. 4. Plot showing Zero-Order Kinetics of DMDS removal for the second column compost mixture (Initial Conc. = 12.8 ppmv, Gas Flow Rate = 36.6 Lpm).

Table 2. Observed zero-Order kinetic coefficients for the first column biofilter.

Inlet Concentration (ppmv)	Residence time (sec)	K_o (ppm/sec)
13.2	52	0.314
29.9	60	0.328
23.6	42	0.334
10.4	60	0.343
46.0	54	0.345
13.7	39	0.379
17.2	50	0.383
23.8	33	0.405
30.4	42	0.411
11.6	60	0.412
25.1	63	0.415
24.0	35	0.420
43.5	54	0.427
17.2	54	0.435
49.6	54	0.436
55.7	54	0.439
25.1	63	0.456
15.4	37	0.459
20.6	54	0.480
19.0	67	0.480
22.8	42	0.486
20.7	54	0.494
29.9	54	0.506
16.9	52	0.513
21.6	60	0.528
49.0	56	0.534
37.1	54	0.545
15.0	50	0.562
15.6	28	0.564
18.6	50	0.620
41.0	54	0.622
29.7	52	0.623
19.8	60	0.629
22.5	40	0.634
20.5	30	0.635
18.8	26	0.639
20.3	54	0.641
19.4	54	0.641
31.7	54	0.648
38.4	56	0.649

biodegradation of dimethyl disulfide followed reaction-limited zero-order kinetics.

Typical zero-order kinetic plots for both the first and second compost mixtures are presented in Figures 3 and 4, respectively. Both compost mixtures showed almost identical average values for zero-order kinetic coefficients. The observed zero-order kinetic coefficients for the first and second compost filters are given in Table 2 and 3, respectively. Average zero-order kinetic coefficient values of 0.50 (s.d. = 0.11, n = 40) ppm/

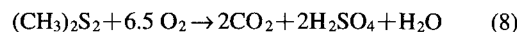
Table 3. Observed zero-Order kinetic coefficients for the second column biofilter.

Inlet Concentration (ppmv)	Residence time (sec)	K_o (ppm/sec)
20.9	55	0.309
39.6	55	0.351
18.2	47	0.360
16.7	49	0.378
10.4	49	0.380
16.9	43	0.381
21.6	61	0.411
27.8	58	0.417
35.7	58	0.434
25.6	43	0.442
12.8	29	0.434
14.5	47	0.451
30.5	58	0.454
12.1	47	0.467
23.6	33	0.493
51.0	55	0.529
38.2	55	0.551
27.5	24	0.581
49.1	55	0.591
45.4	55	0.620
46.1	55	0.638
45.1	55	0.686
23.7	20	0.688
33.7	47	0.703

sec and 0.49 (s.d. = 0.12, n = 24) ppm/ sec were obtained for the first and second compost mixtures, respectively. Variations observed in the individual values of kinetic coefficients presumably were due to changing operating conditions such as acidity, temperature, gas flow, pollutant concentration, etc. The DMDS inlet concentrations studied were in the range from 10 to 55 ppmv. Kinetic studies at lower inlet DMDS concentrations (< 10 ppmv) were not carried out in this work. At the analytical limitations of the gas chromatograph /detector used. At lower inlet concentrations, it is possible that DMDS removal may show first -order macro-kinetics. Wani *et al.* (1998) suggested first-order macro-kinetics for dimethyl sulfide and methyl mercaptan between 0 and 1,000 ppmv concentrations.

3. 2 Microbiological studies

Smith and Kelly (1988a) were the first investigators to report the isolation and breeding of a bacterial strain that was capable of oxidizing DMDS as its sole source of energy. The isolated DMS- and DMDS-oxidizing bacterial specie was grown on thiosulfate-mineral salts agar. This bacterial strain (denoted as strain E6) had an optimum pH in the range from 6.7 to 6.9. Bacterial growth ceased at pH values below 5.5 and above 8.2. Growth studies conducted in liquid batch cultures also revealed an inhibitory DMDS level at approximately 5 mM (close to saturation in aqueous solution). Final biomass levels were proportional to the amount of DMDS supplied and observed sulfate formation correlated with DMDS disappearance and increase in biomass. CO_2 fixation was suggested to be the only route for carbon assimilation during growth on DMDS. No elemental sulfur formation was observed and two moles of sulfate were produced per mole of DMDS. The following stoichiometry for DMDS oxidation was found:



$$(CH_3)_2S_2+6.5\ O_2 \rightarrow 2CO_2+2H_2SO_4+H_2O \quad (8)$$

In a follow-up study, Smith and Kelly (1988b) identified the strain E6 as Thiobacillus thioparus. The strain E6 was morphologically and physiologically an autotrophic, chemolithotrophic Thiobacillus, incapable of growth on organic compounds containing carbon-carbon bonds. Carbon dioxide was fixed through the Calvin cycle, and there was not likely to be any contribution from the serine pathway, that was shown to be essential for growth of the hyphomicrobia on methylated sulfides. Oxidation of DMDS by T. thioparus, strain E6, proceeded by a pathway that was analogous to that reported for the oxidation of methylated sulfides by hyphomicrobia and T. thioparus, strain TK-m. The proposed mechanism for the oxidation of DMDS by T. thioparus, strain E6, is shown in Figure 5. The initial microbiological step in the degradation of DMDS is the reductive cleavage of DMDS to produce two molecules of MM through the use of DMDS reductase. This step is oxygen independent and can be achieved under anaerobic conditions. Using MM oxidase, MM is further broken down to H_2S, hydrogen peroxide (H_2O_2) and formaldehyde (HCHO) under aerobic conditions.

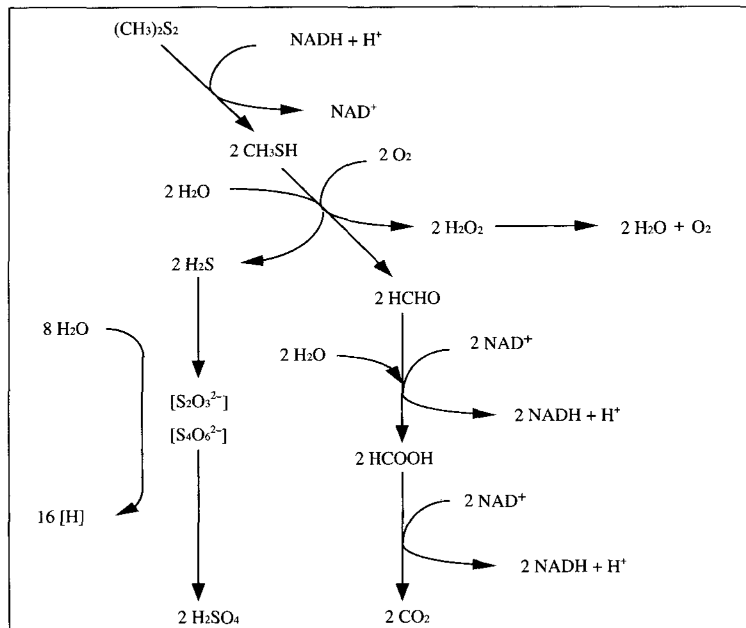


Fig. 5. Proposed pathway for breakdown of dimethyl disulfide by T. Thioparus, strain E6. (Source: Smith and Kelly, 1988b).

The H_2O_2 formed is destroyed by catalase. The latter step is a central one in MM oxidation since it is the site of inhibitory action. H_2S is further oxidized to sulfate through a sulfide-oxidizing system possibly involving thiosulfate and tetrathionate as intermediates. HCHO is converted to formic acid (HCOOH) by formaldehyde dehydrogenase, and HCOOH is converted to CO_2 by formate dehydrogenase enzymes. All of the energy conserved is from the oxidation of HCHO to CO_2, yielding a maximum of four nicotinamide adenine dinucleotide (NADH) per DMDS consumed. Since DMDS reduction initially requires one NADH, only three are available for energy conservation through oxidative phosphorylation. Biomass yield in steady state chemostat cultures growing on DMDS was found to be 14.4 g cell-carbon per mole of DMDS.

As a consequence of decreased efficiencies due to extreme acidification of the filter beds used in this study, methyl mercaptan (MM), an intermediate oxidation product of DMDS, was observed in the gas samples collected. The MM concentrations observed were very low, probably in the ppbv levels. From the peak areas observed on the analytical gas chromatograms, it was concluded that the concentrations of MM decreased progressively along the filter bed, being totally removed at the bed outlet. The MM peak areas increased with inlet DMDS concentrations. This emergence of MM supports the degradation pathway proposed by Smith and Kelly (1988b) for T. thioparus, Strain E6. Hydrogen sulfide, which is also a by-product of DMDS degradation (Smith and Kelly, 1988b) was not observed in the gas samples. This is possibly due to the rapid degradation of H_2S on compost beds (Yang and Allen, 1994).

4. CONCLUSIONS

The macro-kinetic model described by Ottengraf (1986) was used to determine the overall removal kine-

tics of dimethyl disulfide (DMDS). It was found that the biodegradation of DMDS is governed by zero-order reaction-limited macro-kinetics for inlet DMDS concentrations between 10 and 55 ppmv. The overall average zero-order kinetic coefficient for DMDS removal by compost was 0.50 (±0.1) ppm/sec for both compost mixtures studied. Variations in individual kinetic coefficients were observed due to varying environmental conditions, such as pH and temperature. The kinetic coefficients determined are specific to the system discussed in this work. Different values for the zero-order kinetic coefficients may be observed for other systems, using different filter materials, microorganisms, etc. However, in the absence of more specific data the values determined in this work may be used for scale-up studies using certain safety factors. Due to analytical limitations, the macro-kinetics of DMDS concentrations below 10 ppmv could not be quantitatively studied. The macro-kinetics prevailing at these low concentration levels might be different than zero-order kinetics. This omission should be investigated further.

During high acidity conditions in the filter beds, methyl mercaptan (MM) was observed in the gas samples collected. Appearance of MM was probably due to decreased microbial activity in the lower portions of the biofilter. The presence of MM in the gas samples verified the findings of Smith and Kelly (1988b) that MM is an intermediate in the degradation of DMDS. Considering the neutral pH range required and the presence of MM, it is likely that the microorganisms present in the biofilters used in this research are similar to the T. thioparus (strain E6) species reported in the literature.

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