THE IMPACT OF OZONATION AND BIOLOGICAL TREATMENT ON DISINFECTION BY-PRODUCTS

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INTRODUCTION

Disinfection of drinking water leads to the formation of disinfection by-products (DBPs). Organic and inorganic DBPS are formed by the reaction of the disinfectant with natural organic matter (NOM) and with inorganic compounds, respectively. Some of these DBPs are regulated or are being considered for regulation because of the health concern associated with them. In the seventies, trihalomethanes (THMs) were the first group of DBPs that were regulated with a maximum contaminant level (MCL) of 100 μg/L. This MCL may be reduced in the near future and MCLs may be set for the individual species.

In an effort to minimize DBP formation while still providing microbially safe water, modifications in drinking water treatment have become necessary. These include the use of alternative disinfectants and more efficient DBP precursor removal.

Microbial regrowth in the distribution system can be controlled by limiting nutrient availability and by the presence of residual disinfectant. Ozonation often increases the biodegradable organic carbon in the water which would serve as a nutrient for the microorganisms. (Langlais et al., 1991). Aldehydes and other oxidation by-products are among these biodegradable ozone DBPs. Ozone also reacts with bromide and generates bromate, an inorganic DBP that may be regulated in the future.

In the presence of bromide, the halogenated DBPs are a mixture of brominated and chlorinated products. Ozone or chlorine can oxidize bromide to form free bromine which can react with NOM. Typically, the speciation shifts to the more brominated DBPs (Amy et al., 1991) as the rate of bromine substitution is believed to be faster than chlorine substitution (Symons et al., 1987). Bromate is formed by the further oxidation of hypobromite.

Ozonation used in drinking water treatment does not seem to significantly affect the organic carbon concentration, but does change the nature of the precursor compounds. The concentration of halogenated DBPs formed by subsequent chlorination is often decreased when compared

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to that formed with no pretreatment (Reckhow and Singer, 1984, Shukairy and Summers, 1992a, Miltner et al., 1992). Upon ozonation, the bromide concentration decreases because of the formation of bromate, and consequently the decrease in the ratio of bromide to organic matter leads to a shift in DBP speciation to the more chlorinated products.

The use of biotreatment in United States drinking water treatment plants is very limited. Biological treatment affects the nature of NOM. The dissolved organic carbon (DOC) is decreased and the reactivity to subsequent chlorination may be decreased as a consequence of the oxidation of the precursor compounds. Biotreatment downstream of ozonation may biodegrade some of the ozone DBPs and chlorine DBP precursors and minimize substrate availability for regrowth (Miltner and Summers, 1992). A review of the use of biotreatment for the control of DBPs is given by Shukairy and Summers (1992b).

Biotreatment reduces the DOC concentration, while the bromide concentration remains constant, resulting in an increase in the bromide to DOC (Br/DOC) ratio. An increase in Br/DOC has been shown to shift the speciation to the more brominated DBPs (Amy et al., 1991). Upon subsequent chlorination, DBP formation and speciation seem to be a function of: DOC, oxidation of the precursor compounds, bromide concentration and the chlorination conditions that are used, i.e. pH, temperature, holding time and the chlorine dose. At high chlorine to bromide ratios, the speciation shifts to the more chlorinated byproducts.

OBJECTIVES

This paper summarizes several recent studies on the impact of ozonation and biological treatment on DBP formation. Ozonation was characterized by the formation of ozonation DBPs such as aldehydes, assimilable organic carbon (AOC), biodegradable dissolved organic carbon (BDOC) and the oxidation of bromide to bromate. The oxidation of the DBP precursor compounds and the effectiveness of biotreatment for the control of DBPs were monitored by the formation potential (FP) for total organic halogen (TOX), total THMs (TTHMs) and total measured haloacetic acid (THAAs). Special attention was also given to the impact of bromide concentration on the speciation of the DBPs.

A specific objective of this paper was to compare DBP control by batch biological treatment at the bench-scale to that by a continuous flow sand filter at the pilot-scale.

EXPERIMENTAL APPROACH

Raw Ohio River water was used in both the bench and pilot-scale biotreatment studies. Ozone was applied in a pilot-scale countercurrent reactor in both studies. For the bench-scale biotreatment, the experimental design was set up as a 6 by 3 by 2 matrix. Ozone was dosed at five selected levels and a control. Three bromide levels, 50.7 μg/L (ambient), 258, 550 μg/L, were selected. Each ozone - bromide combination was run with and without biotreatment. Biotreatment was carried out in mixed batch reactors. The water quality parameters examined were: chlorine demand,

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DOC, BDOC, AOC, spectral absorption coefficient (SAC), bromide and bromate, aldehydes, TOX , THMs and HAAs.

In comparison, for the pilot-scale biotreatment, raw Ohio River water was ozonated at an ozone to DOC ratio of 0.7 mg/mg. After coagulation, flocculation and settling, the flow was split to eight parallel filters. (Miltner and Summers, 1992). The results from a fixed bed single pass filter with sand will be reported in this paper.

MATERIALS AND METHODS

Raw Ohio River water was trucked from the Cincinnati Water Works to the USEPA facility. Ozonation was conducted at room temperature in a 15 cm (6 in.) diameter countercurrent flow-through contactor. The transfer efficiency was greater than 94 percent. This ozonation system has been described in detail (Miltner et al., 1990). After each change in ozone dose, samples were collected after steady state conditions were reached, which was conservatively chosen at two times the T_{100} time based on tracer studies. Applied gas to the contactor and off-gas from the contactor were measured by UV. Dissolved ozone from the contactor was measured spectrophotometrically using the indigo trisulfonate method (Bader and Hoigne, 1981). For the bromide spike, a concentrated stock potassium bromide solution was added to the raw water before ozonation. To provide the required concentration as bromide, the rate of flow of the stock solution was varied. Samples were collected before and after contact with ozone.

For the pilot-scale system, ozonation, alum coagulation, flocculation, sedimentation and sand filtration were used. The sand filter (Filter 5), was a 3.8 cm (1.5 in.) diameter glass column containing 76 cm (30 in.) of sand (ES= 0.45 mm) supported by 20 cm (8 in.) of gravel and 10 cm (4 in) of coarse sand.

For the bench-scale biotreatment, batch reactors were used. The reactor is a modification of the Joret-Levi reactor (Joret et al., 1988). To provide enough sample for formation potential determination, a 2 L Erlenmeyer flask containing 300 ml of Ohio-River-water-acclimated-sand was used with a sample volume of 1 L. Vacuum was applied to the reactor forcing the incoming air through two water traps for scrubbing and humification. This air was to provide enough oxygen for the bacterial growth and for mixing. The samples were kept in the bioreactors for five days, and before formation potential tests, were filtered using prewashed 0.45 μm pore diameter (Millipore HV) membrane filters. Biodegradation was measured by the difference between the DOC before and after biotreatment.

DOC was measured by EPA Method 415.1 and UV absorbance was measured at 254 nm and reported as SAC. AOC was measured using the methods of van der Kooij et al. (1982,1987). Two strains of heterotrophs, Pseudomonas fluorescens P17 and Spirillum NOX, were employed in AOC determinations. Bromide and bromate concentrations were measured by ion chromatography by the method of Hautman and Bolyard (1991). The minimum detection level for bromate was approximately 7 μg/L. Aldehyde concentrations were

measured by a modification of the PFBOA derivitization method described by Miltner et al. (1991).

Samples were collected for formation potential tests before and after ozonation and after biotreatment. For formation potential determination, a 12 mg/L chlorine dose was used, at ambient pH (7.5-8.0), at 20 °C and held headspace-free in the dark for seven days. After quenching, samples were collected for DBP analyses that were based on USEPA methods: THMs were measured by EPA Method 551; TOX by EPA Method 450.1 and HAAs by EPA Method 552.

For quality assurance ten percent duplication was used. Error bars in the figures represent the range of results from the duplicate biotreatment or ozonation tests. Error bars for biotreated samples represent the range of results from two separate reactors.

RESULTS AND DISCUSSION

The experimental matrix of the bench-scale biotreatment study is summarized by the DOC results in Figure 1 which show the impact of ozonation and biological treatment on DOC concentration. For all bromide levels, ozonation did not have a significant impact on the DOC concentration. Biological treatment of the raw water (O_3/DOC ratio equal to 0 mg/mg) resulted in a 13 to 14 percent removal of DOC. With ozonation followed by biotreatment, a range of 20 to 40 percent DOC removal was observed, with the majority of the effect occurring at O_3/DOC ratios less than 0.8 mg/mg.
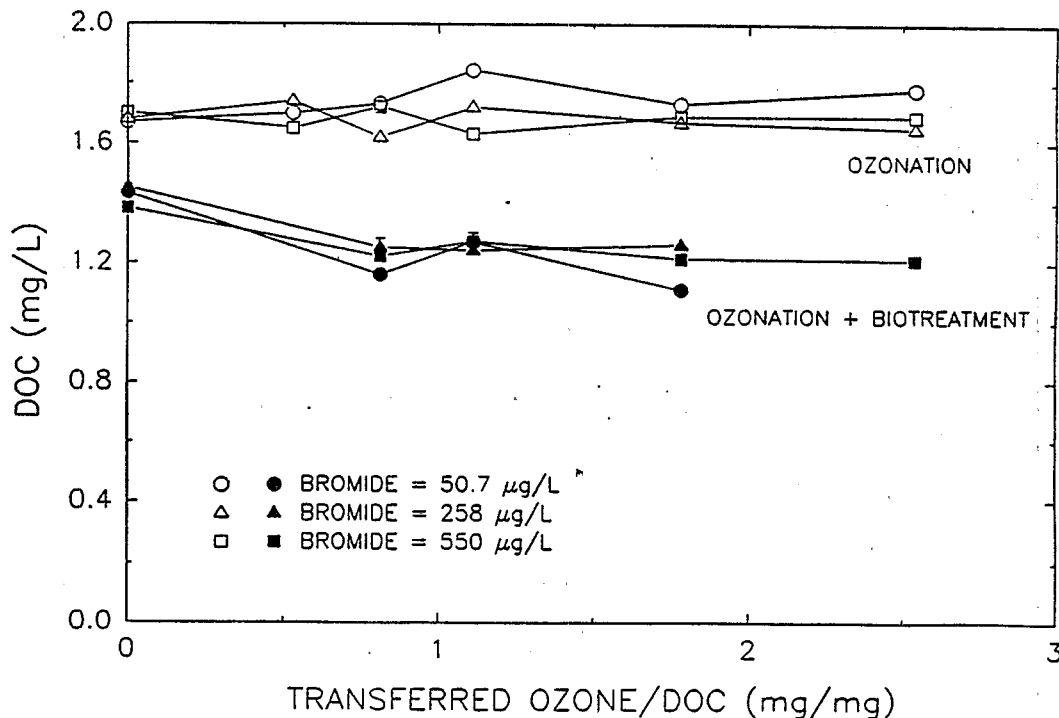

Figure 1. BENCH-SCALE BIOTREATMENT: IMPACT OF OZONATION AND BIOTREATMENT ON DOC CONCENTRATION AT THREE BROMIDE LEVELS.

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Biological treatment not only removed the DOC but also seemed to lower the chlorine demand (Figure 2). In this case, ozonation alone enhanced chlorine demand removal, biotreatment alone decreased the demand by 43 percent, and the combination of ozonation and biotreatment decreased the demand by a range of 60 to 70 percent.
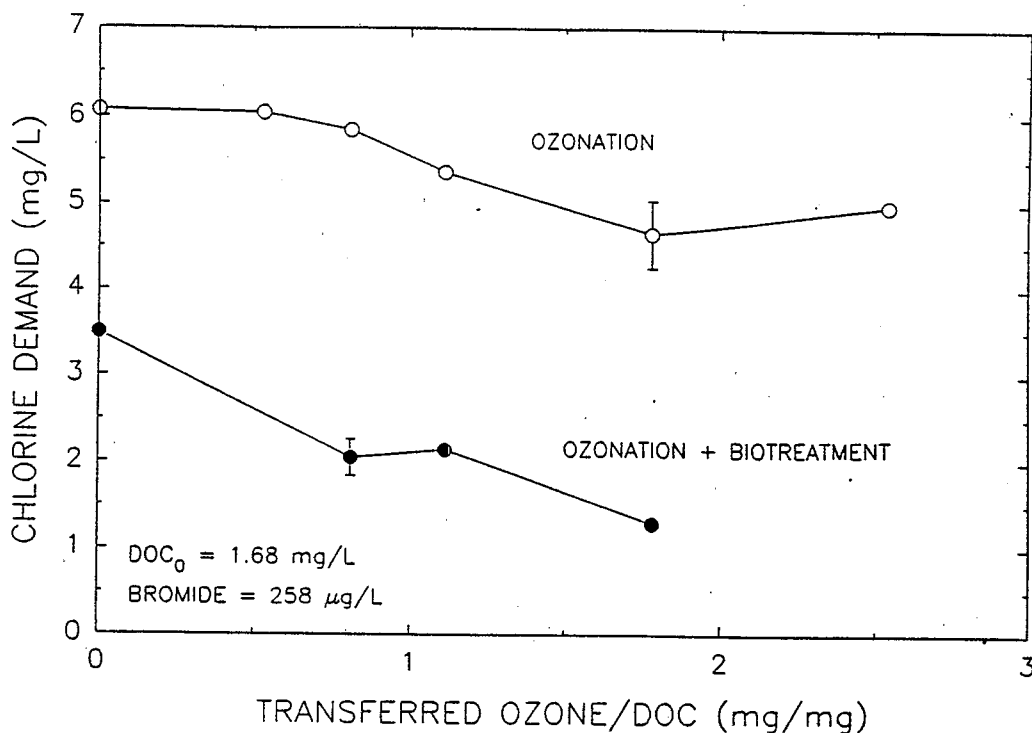

Figure 2. BENCH-SCALE BIOTREATMENT: IMPACT OF OZONATION AND BIOTREATMENT ON CHLORINE DEMAND.

The halogenated DBPs were also decreased by ozonation and by biotreatment. The total THM formation potential results are presented in Figure 3. Ozonation decreased the TTHMFP by a range of 27 to 46 percent, and a 28 percent reduction in TTHMFP was observed by biotreatment alone. In comparison, at all ozone doses, the effect of biotreatment was another reduction of 12 to 19 percent. TOXFP and total measured HAAFP results from this study, though not shown, were also decreased by ozonation and by biotreatment (Shukairy et al., 1992b).

Ozonation DBPs
BDOC and AOC: The formation of biodegradable organic matter by ozonation is shown in Figures 4 to 7. The BDOC increased with increasing ozone doses at all bromide levels, Figure 4. The most significant increase in BDOC was at O_3/DOC ratios less than 0.8 mg/mg. At higher ozone doses, the increase in BDOC was minimal. Similar site specific BDOC behavior at higher ozone doses have been reported (Langlais et al., 1991). AOC measured by both strains NOX and P17 increased with increasing ozone dose as
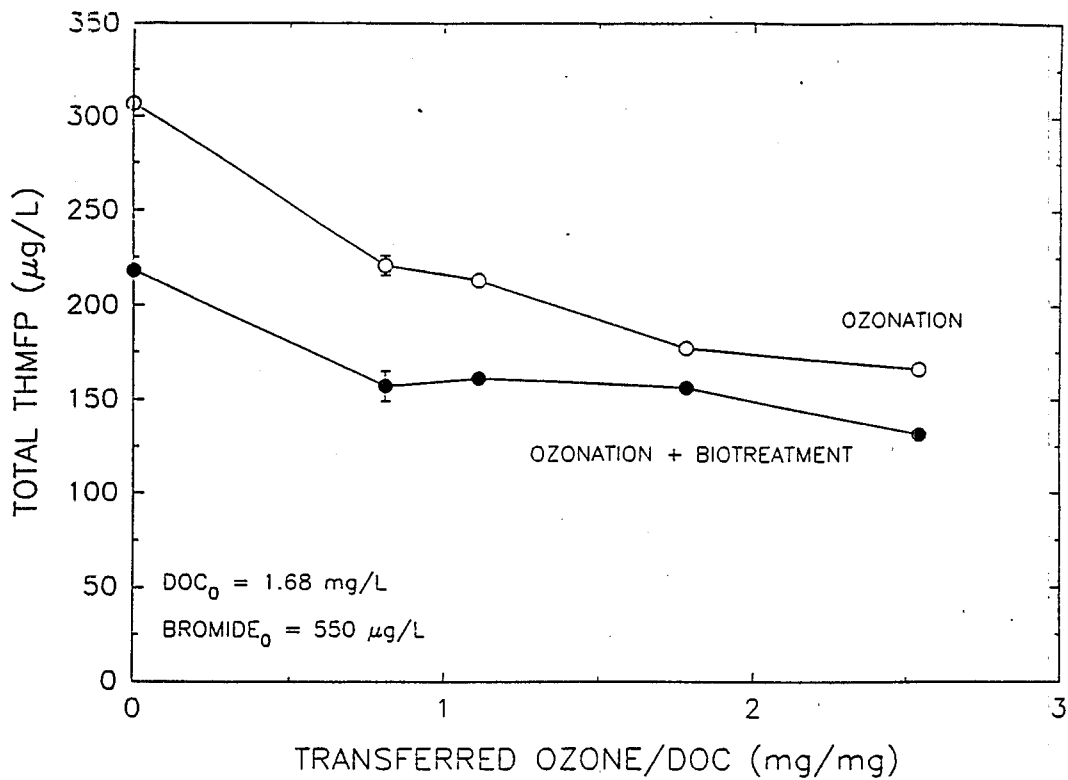
Figure 3. BENCH-SCALE BIOTREATMENT: IMPACT OF OZONATION AND BIOTREATMENT ON TOTAL THM FORMATION POTENTIAL.
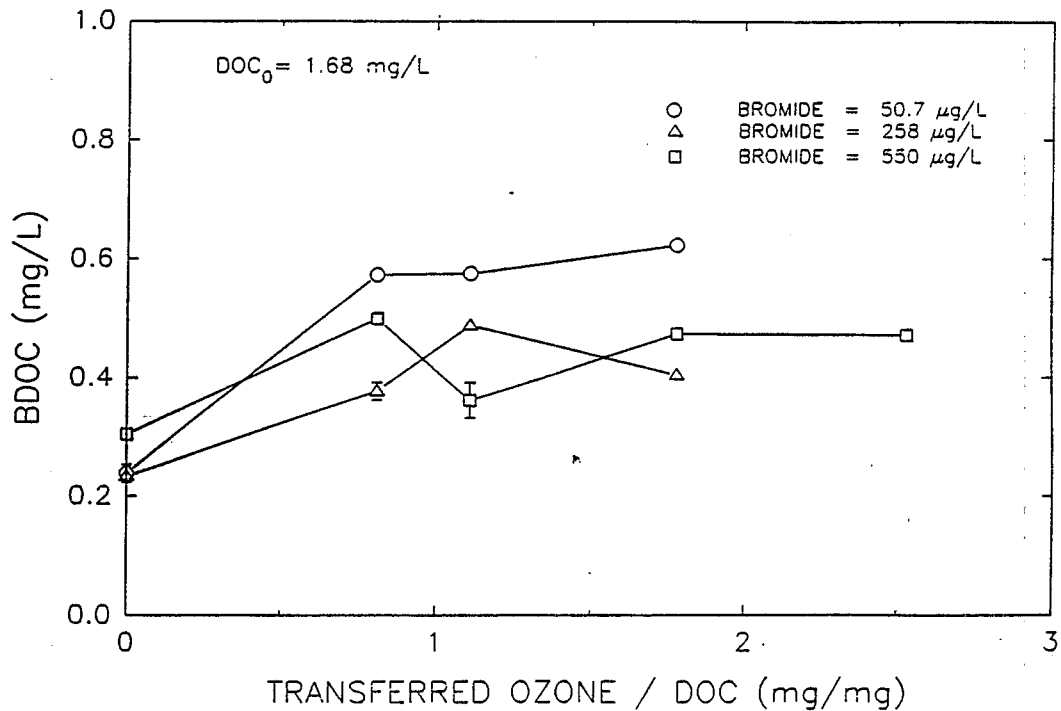

Figure 4. BENCH-SCALE BIOTREATMENT: IMPACT OF OZONATION ON BDOC AT THREE BROMIDE LEVELS.

shown in Figure 5. Most of this increase occurred at O_3/DOC ratios below 1.2 mg/mg. At higher ozone doses, AOC increases were minimal.
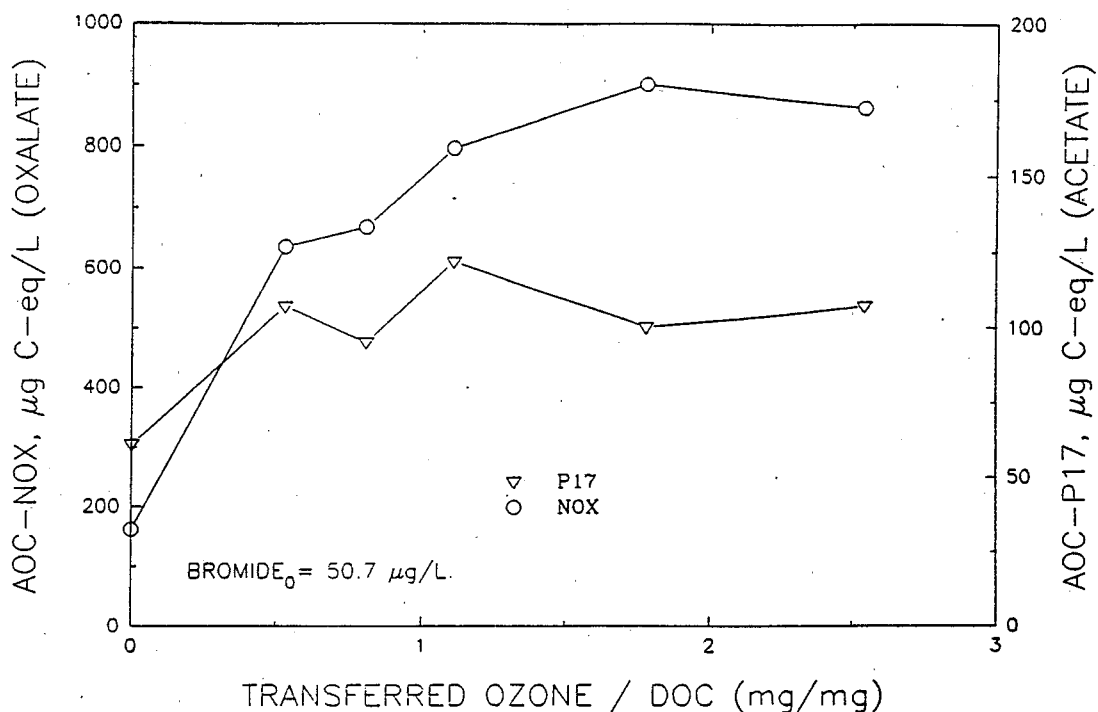

Figure 5. IMPACT OF OZONATION ON AOC (P17 AND NOX).

In a long-term pilot study, increase in BDOC and AOC were similar to that described in Figures 4 and 5. Raw water BDOC levels increased an average of 70 percent upon ozonation (O_3/DOC approximately 0.7 mg/mg) to the levels described in Figure 6. Raw water AOC-NOX levels increased an average of 390 percent upon ozonation.

Both measures of bacterial nutrients were lowered during sedimentation, suggesting that suspended bacteria can biodegrade these compounds during the 5 and a 1/2 hour sedimentation time. This is illustrated for BDOC in Figure 6.

In biological sand filters, both were readily biodegradable. Figure 6 shows control of BDOC in Filter 5; Figure 7 shows control of AOC-NOX in Filter 5. Figure 7 illustrates that about 30 days were required to establish bioactivity in Filter 5 as no control was observed before that time; insufficient BDOC samples were collected in that time to determine the time required to establish its control.
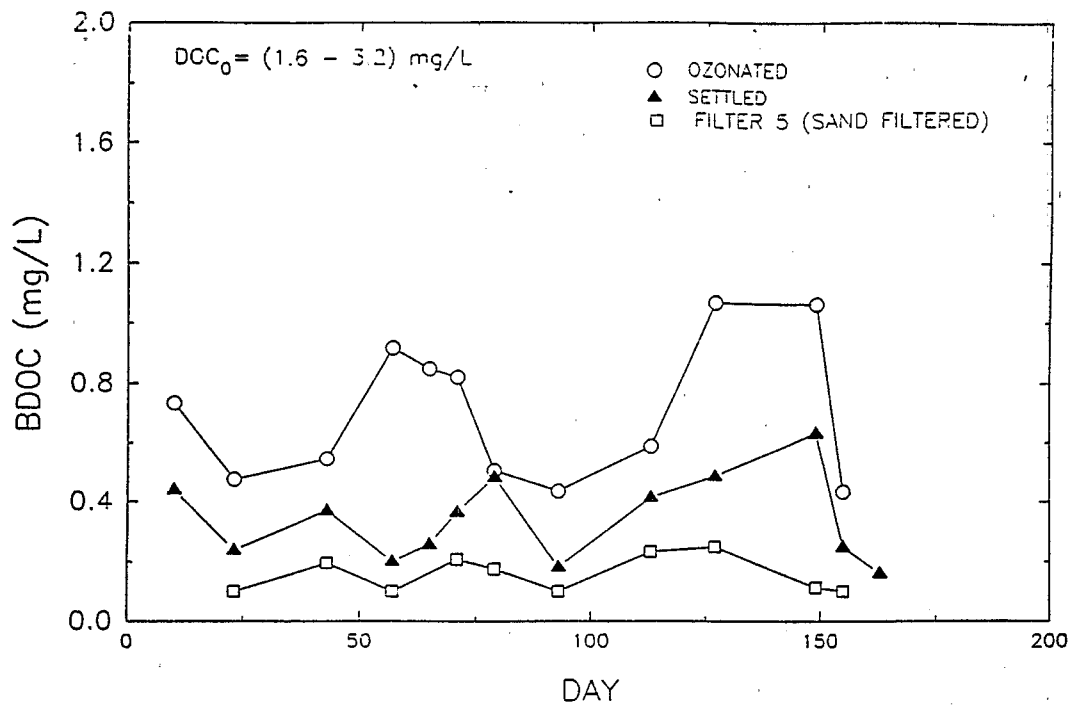
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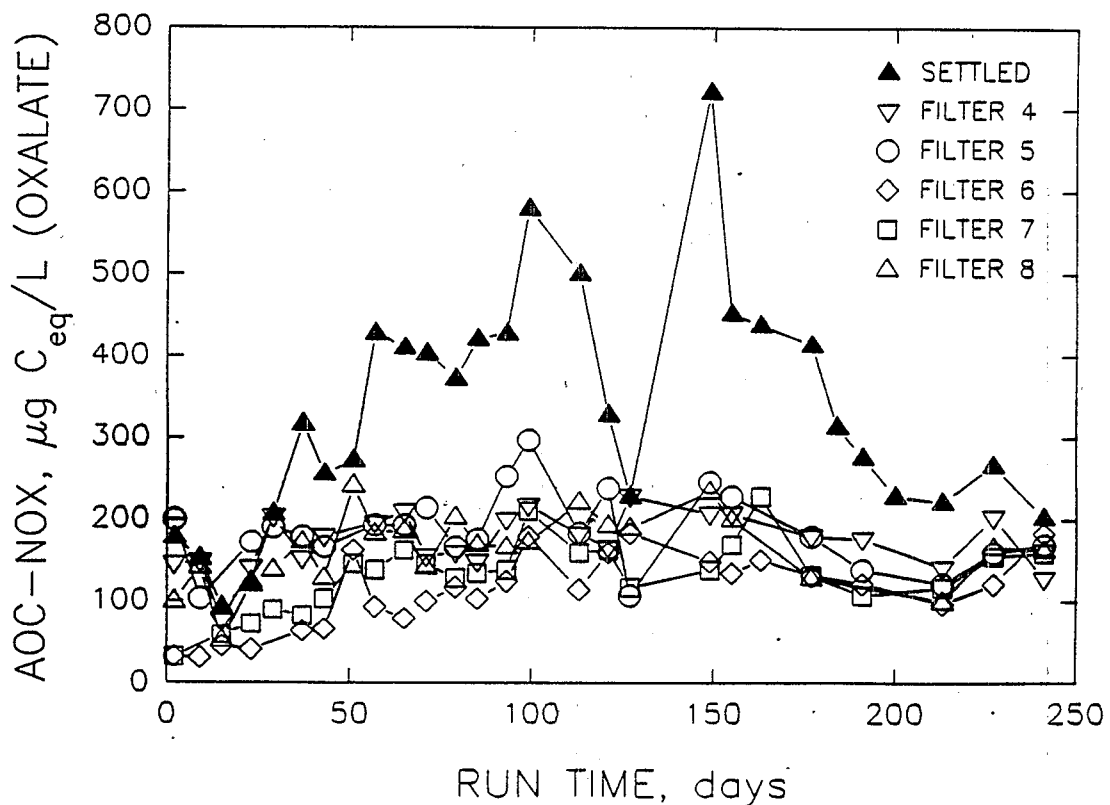**Figure 6. PILOT-SCALE BIOTREATMENT: IMPACT OF OZONATION AND SAND FILTRATION ON BDOC.**

Figure 7. PILOT-SCALE BIOTREATMENT: CONTROL OF AOC-NOX IN BIOLOGICAL FILTERS.

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Aldehydes: An increase in aldehyde concentrations with ozonation of Ohio River water is shown in Figure 8. All aldehydes detected, methyl glyoxal, propanal, pentanal, acetaldehyde and methyl glyoxal, were subsequently reduced by biotreatment, at all ozone doses, to concentrations less than that in the raw water. Similar decreases in aldehyde concentrations attributed to biotreatment have been reported (Shukairy et al., 1992a; Miltner et al., 1990), indicating that these simpler molecules are very biodegradable.
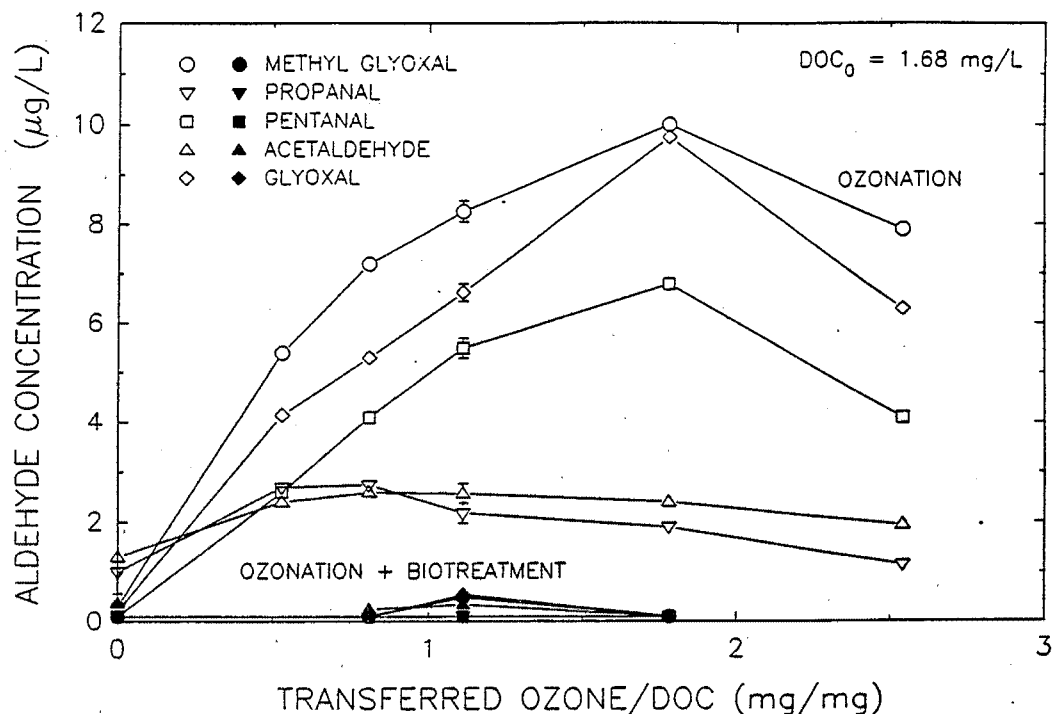

Figure 8. BENCH-SCALE BIOTREATMENT: IMPACT OF OZONATION AND BIOTREATMENT ON THE FORMATION AND CONTROL OF ALDEHYDES.

The biodegradability of aldehydes in the pilot-plant is also apparent from the behavior of these compounds in the sand filter (Filter 5). Figure 9 shows the results for methyl glyoxal. For the control of this aldehyde, little or no time was required to bioacclimate these filters as removal was near 100 percent in the first week of operation. The other aldehydes examined but not shown, formaldehyde, glyoxal and acetaldehyde, were not as biodegradable as methyl glyoxal but the trends in their control were similar to that of methyl glyoxal.
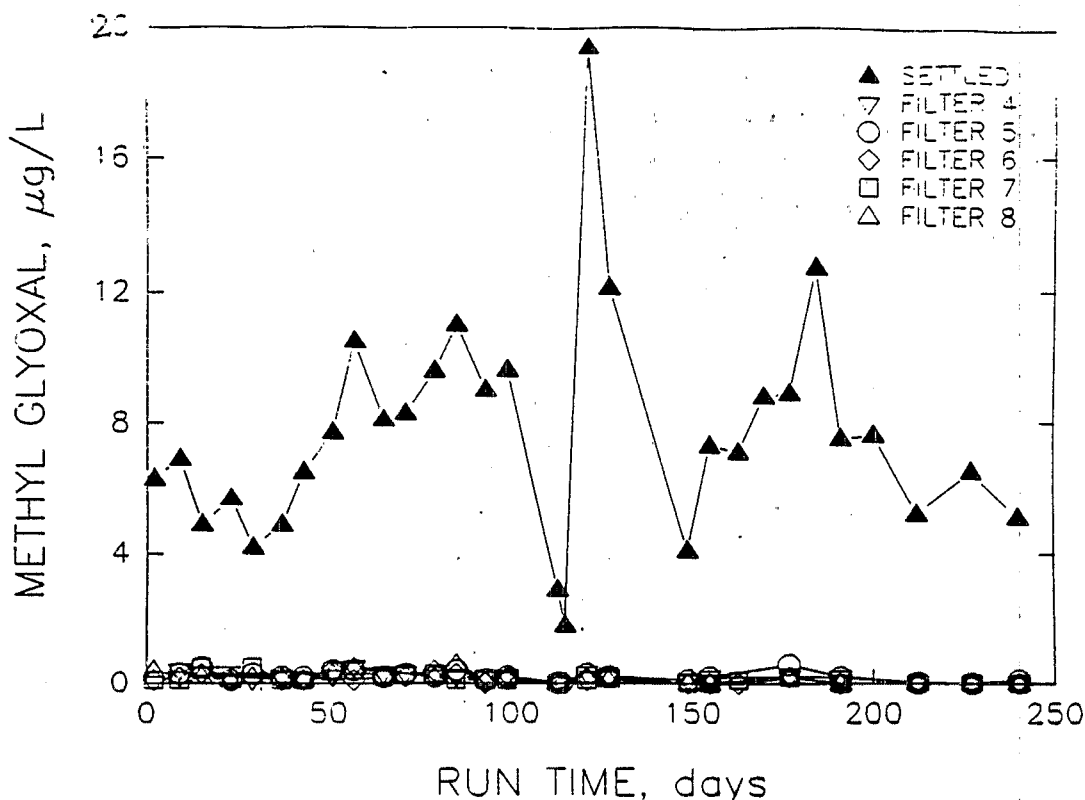
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Figure 9. PILOT-SCALE BIOTREATMENT: CONTROL OF METHYL
GLYOXAL IN BIOLOGICAL FILTERS.

Bromate: In the presence of bromide, ozone oxidizes
the bromide to hypobromite ion and then to bromate. At
ambient bromide concentrations, 50.7 μg/L, bromate was not
detected at O_3/DOC ratios less than 0.8 mg/mg. Increasing
ozone doses caused bromate levels to increase while the
bromide concentration decreased simultaneously (Figure 10).
At the highest O_3/DOC ratio studied, 2.54 mg/mg, 50 percent
of the initial bromide concentration had reacted to form
bromate. At the highest bromide level investigated, 550
μg/L, bromate was detected at the lowest ozone dose in this
study and showed continued increase with increasing ozone
dose to 300 μg/L, Figure 11. At this highest dose studied,
34 percent of the initial bromide concentration had reacted
to form bromate.

DBP Speciation
    The presence of bromide in the water affects the
speciation of the DBPs. Upon ozonation, the bromide
concentration decreased due to bromate formation (Figures
10 and 11). The DOC of the water was not significantly
altered however (Figure 1). This led to a significant
decrease in the Br/DOC ratio. Figure 12 shows the impact
of ozonation and biotreatment on the Br/DOC ratio. At all
bromide levels, the ratio decreased by ozonation compared
to the raw water by as much as 50 percent.

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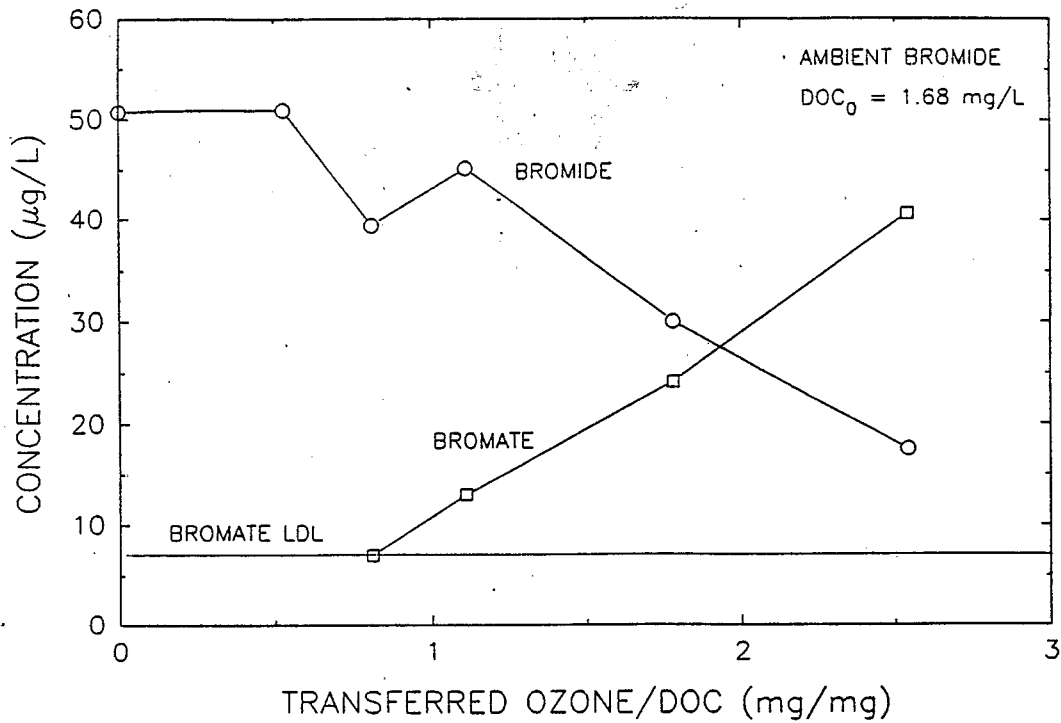
Figure 10. CONVERSION OF BROMIDE TO BROMATE WITH OZONATION AT THE AMBIENT BROMIDE LEVEL.
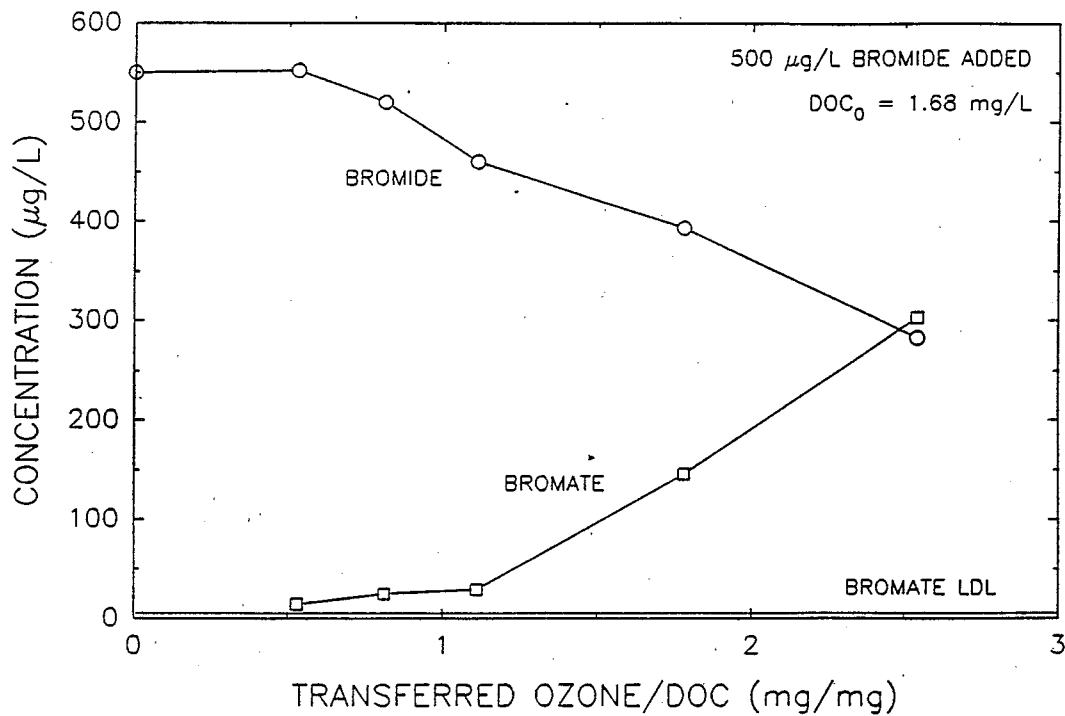
Figure 11. CONVERSION OF BROMIDE TO BROMATE BY OZONATION AT A TOTAL INITIAL BROMIDE CONCENTRATION OF 550μg/L.

With biotreatment alone, the DOC decreased significantly (Figure 1), while the bromide concentration remained unchanged. Therefore the Br/DOC ratio increased as shown in Figure 12. The impact of bromate formation at high ozone doses was also apparent in the biotreated samples. The Br/DOC ratio decreased with increasing ozone dose as compared to the raw biotreated water. In both ozonated and unozonated samples, biotreatment increased the Br/DOC ratio in comparison to the nonbiotreated sample.
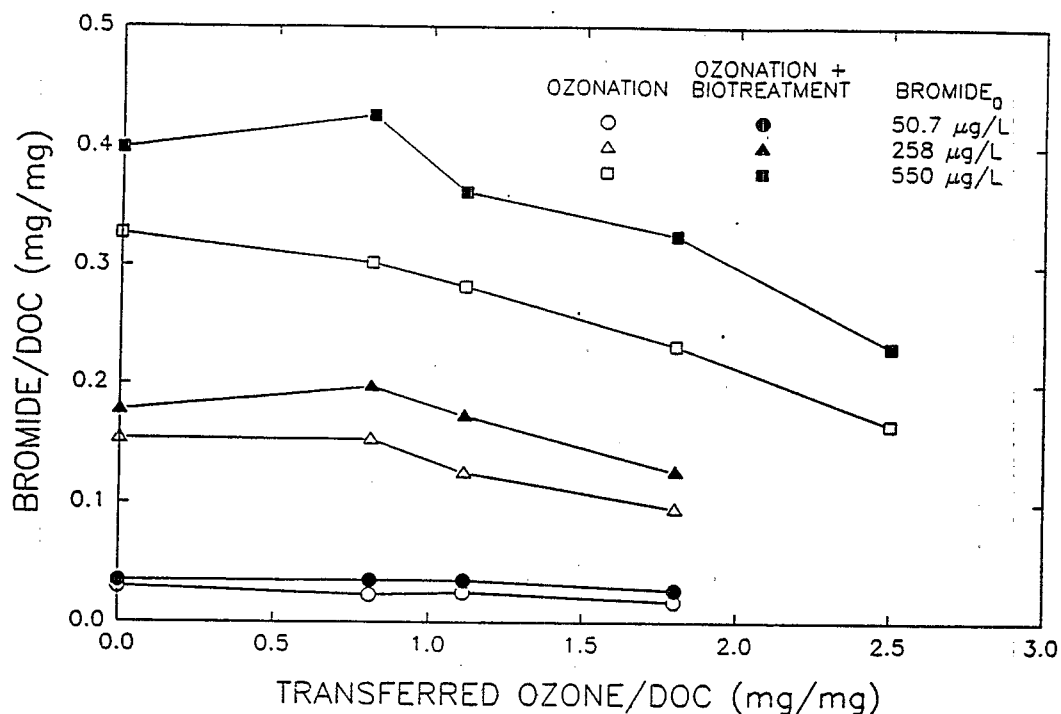

Figure 12. BENCH-SCALE BIOTREATMENT: IMPACT OF OZONATION
AND BIOTREATMENT ON THE BROMIDE TO DOC RATIO.

The change in Br/DOC ratio is very important as it affects the speciation of the DBPs. As the ratio increases, a shift to the more highly brominated species occurs. Figure 13 and 14 show the impact of bromide addition on THM and HAA speciation, respectively, for the raw untreated Ohio River water. Chloro-substituted species formation decreased as the Br/DOC ratio increased, while the more bromo-substituted species increased. At the lowest Br/DOC ratio (50.7 μg/L Br), chloroform was the dominant species and was decreased by 80 percent at the highest bromide concentration studied. At the highest Br/DOC ratio, the dibromochloromethane ($CHClBr_2$) became the most dominant species, and chloroform the least. Bromoform increased significantly with increasing Br/DOC (Figure 13). For HAA species, Figure 14, dichloroacetic acid (DCAA) was the most dominant species at low Br/DOC ratios and decreased by 71 percent as the ratio increased. At the
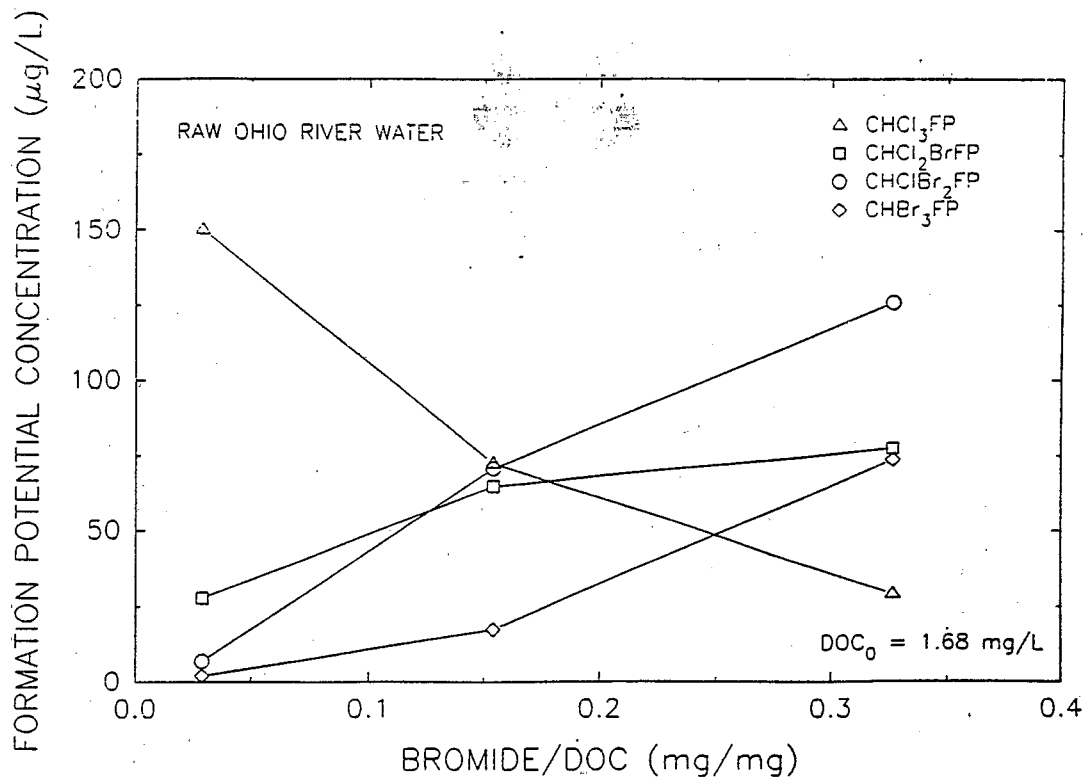
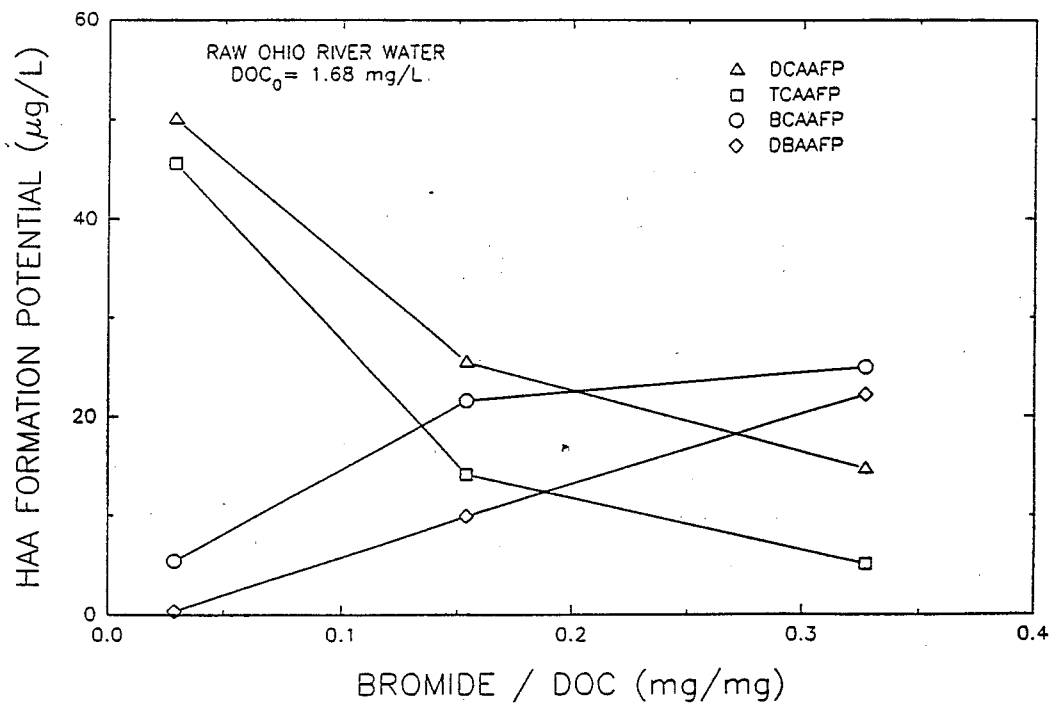**Figure 13. IMPACT OF BROMIDE ADDITION ON THM SPECIATION OF UNTREATED WATER.**

Figure 14. IMPACT OF BROMIDE ADDITION ON HAA SPECIATION FOR UNTREATED WATER.

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highest Br/DOC ratio, bromochloroacetic acid (BCAA) became the most dominant species.

This shift in speciation would also be more significant in full distribution systems or in simulated distribution system studies where the chlorine dose would be much lower than in these FP studies. At low chlorine to bromide ratios the speciation is more shifted to the bromo-substituted species.

The impact of the change in Br/DOC ratio by ozonation and biotreatment on DBP speciation can also be inferred from the individual species behavior. Figure 15 shows the control of formation potential by ozonation and biotreatment of chloroform ($CHCl_3$) and bromodichloromethane ($CHCl_2Br$) at ambient bromide. The percent removal is calculated relative to the concentration of the particular species in the control sample, i.e. the raw water. For this study, chloroform represents 80 percent of the TTHMs and its behavior is similar to that of the TTHMs. Oxidation of the precursor compounds by ozonation resulted in 22 percent removal of $CHCl_3FP$. This removal was not enhanced by increasing ozone dose. Similar behavior upon ozonation was observed for $CHCl_2BrFP$. With biotreatment, DOC removal and biological oxidation of the precursors resulted in improved removal of $CHCl_3FP$, 30 percent for the raw water and up to 50 percent by ozonation and biotreatment.
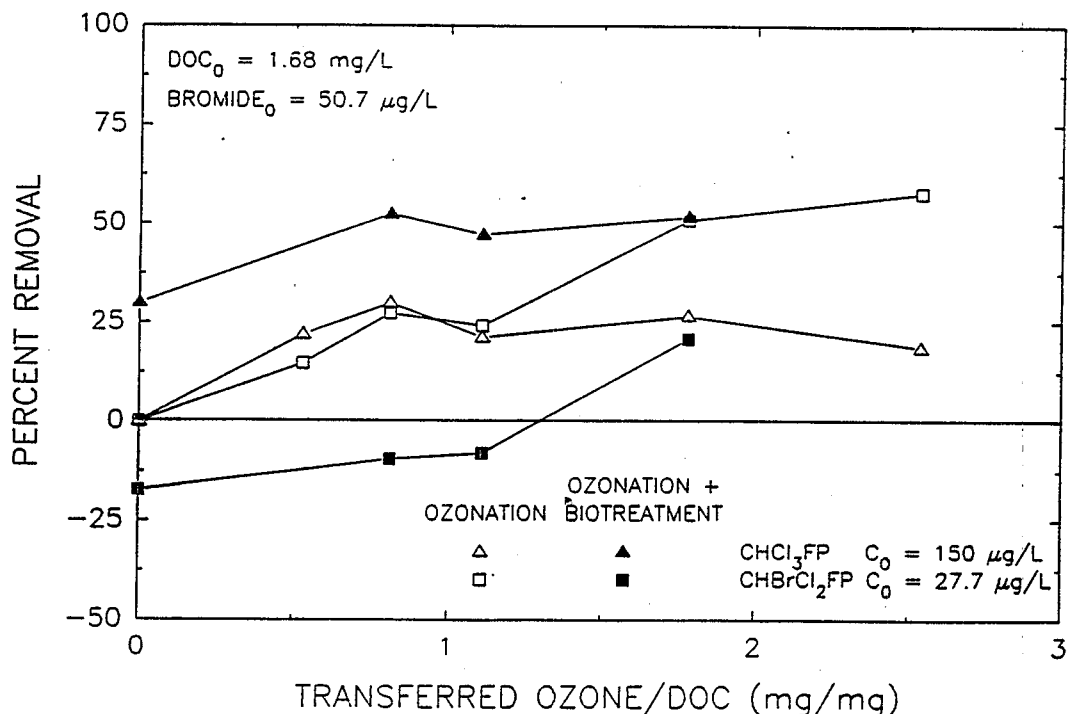

Figure 15. BENCH-SCALE BIOTREATMENT: PERCENT REMOVAL OF THM SPECIES BY OZONATION AND BIOTREATMENT.

The behavior of bromodichloromethane upon biotreatment was slightly different however. An increase in the Br/DOC ratio due to the decrease in DOC with biotreatment resulted in 17 percent increased formation of $CHCl_2Br$. As the bromide concentration decreased, with increasing ozone dose, because of bromate formation (Figure 10), the Br/DOC ratio decreased resulting in removal of $CHCl_2BrFP$.

The more highly brominated DBP species displayed behavioral trends very different from that of the chlorinated DBPs. For dibromochloromethane, at the ambient bromide concentration and at an O_3/DOC ratio of 0.53 mg/mg, there was an increase of 26 percent in formation as shown in Figure 16. Bromate was not detected at this ozone dose (Figure 10), hence the bromide was oxidized to form free bromine and react with the organic matter upon chlorination. As the ozone doses increased, bromate concentrations increased, bromide decreased, the Br/DOC ratio decreased and the speciation shifted to the more chloro-substituted species (59 percent decrease in the $CHBr_2ClFP$ at an O_3/DOC ratio of 2.54 mg/mg).

With biotreatment at ambient bromide levels, the Br/DOC ratio was even higher than in the raw water leading to the formation of $CHBr_2Cl$. The trend upon ozonation/biotreatment was similar to the trend observed for ozonation only. A 25 percent increase in the formation of $CHBr_2Cl$ was observed at an O_3/DOC ratio of 1.78 mg/mg.

For bromoform, the behavior was similar to that of $CHBr_2Cl$ upon ozonation. With biotreatment however, bromoform was removed at all ozone doses. As the organic matter concentration was significantly decreased by biotreatment bromoform was no longer detected.
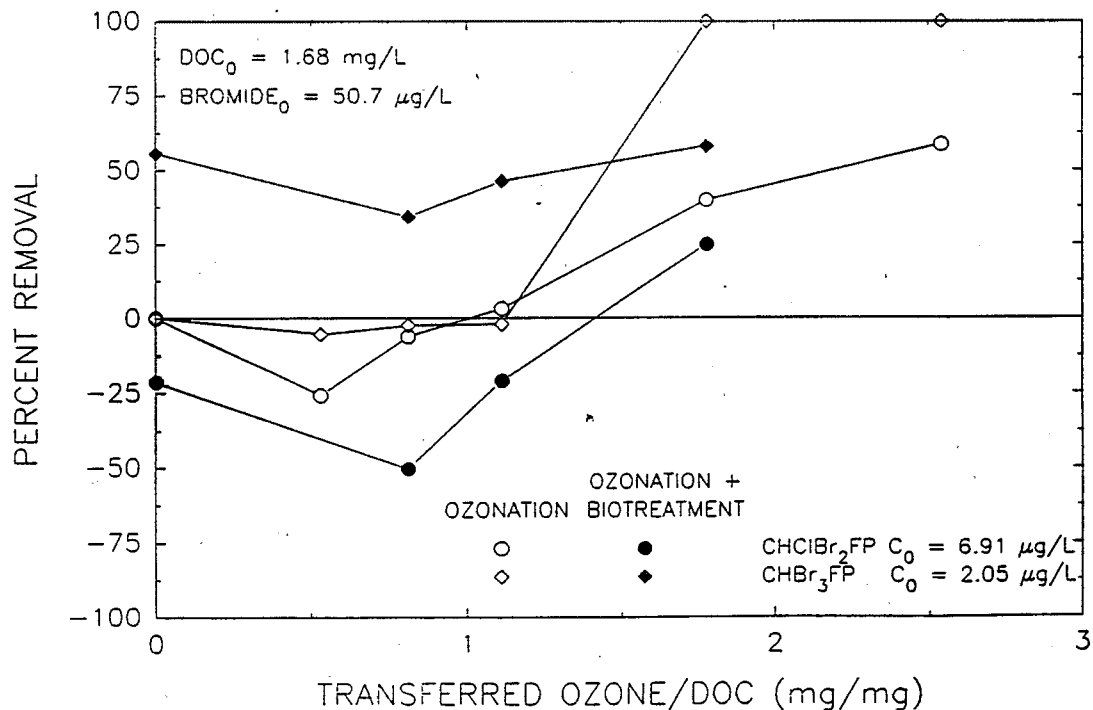

Figure 16. BENCH-SCALE BIOTREATMENT: PERCENT REMOVAL OF THM SPECIES BY OZONATION AND BIOTREATMENT.

HAA speciation trends, similar to those of the THMs, were observed at all bromide levels. Figure 17 shows the percent control of DCAAFP and DBAAFP at ambient bromide. Similar behavior was also observed for TCAAFP. The highly chlorinated HAA species responded to ozonation and biotreatment in a manner similar to those of the highly chlorinated THM species.

Low ozone doses resulted in 16 to 18 percent removal of DCAAFP, and biotreatment, with and without ozonation, resulted in improved removal (50 to 70 percent). For the DBAA species, at a low O_3/DOC ratio there was 35 percent increase in formation. As the ozone dose increased however, the bromide concentration decreased and a 100 percent removal of DBAAFP was observed at an O_3/DOC ratio of 2.54 mg/mg. This effect was even more pronounced with biotreatment.

In comparison, on a pilot-scale for Filter 5, the behavior of DCAAFP is shown in Figure 18. Filter 5 averaged 41 percent removal of DCAAFP when steady state conditions were reached. It took near 50 days to establish steady state removal of DCAAFP in this sand filter. Generally it took one and a half to two and a half months to establish steady state biodegradation of precursors for THMs, HAAs and TOX. This is longer than the time required to establish control of aldehydes (only days) or AOC-NOX (one month). Precursors of halogenated compounds are larger molecules than the smaller, more biodegradable ozone DBP molecules.
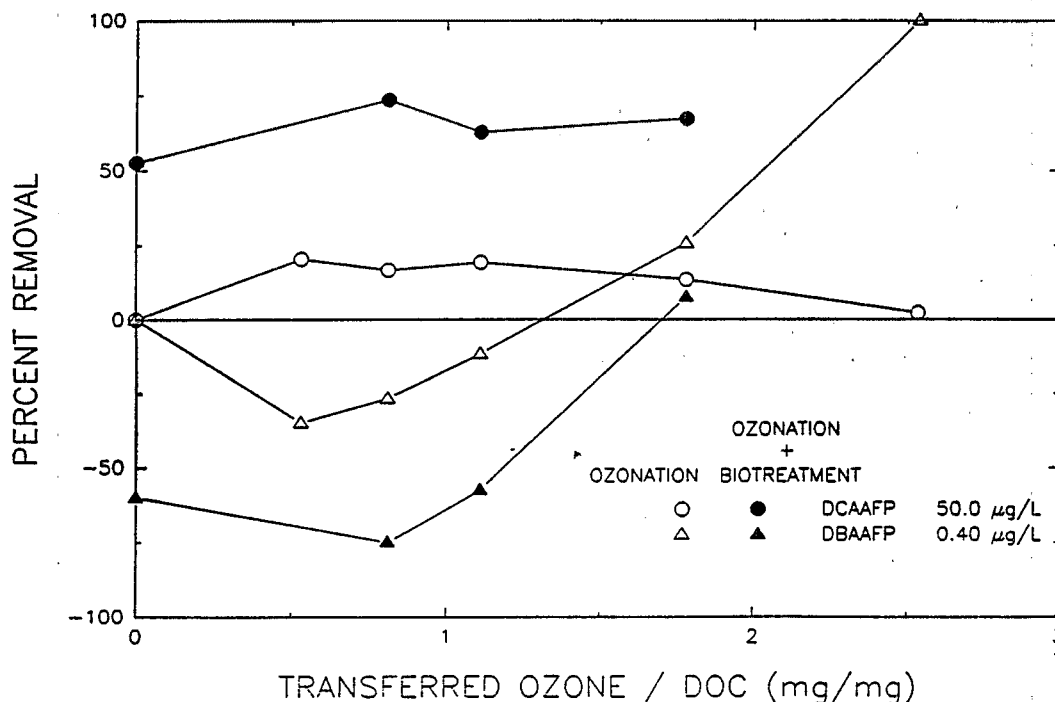

Figure 17. BENCH-SCALE BIOTREATMENT: PERCENT REMOVAL OF HAA SPECIES BY OZONATION AND BIOTREATMENT.
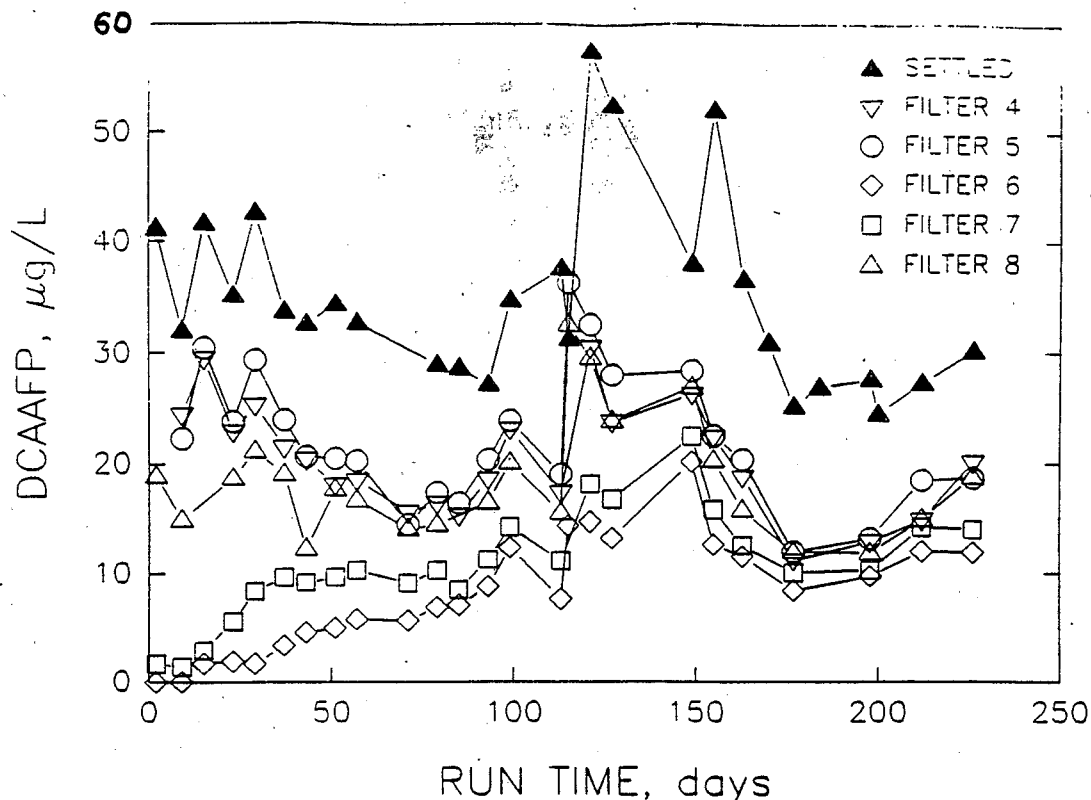
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Figure 18. PILOT-SCALE BIOTREATMENT: CONTROL OF DCAAFP IN BIOLOGICAL FILTERS.

Bench-Scale versus Pilot-Scale Biodegradation

The bench-scale bioreactors used in this study provide five day of contact time at 20 °C for water and bioacclimated sand surfaces. Mogren et al. (1990) and Joret et al. (1988) found five days sufficient to provide optimum biodegradation. In these studies that provided greater than 95 percent biodegradation of aldehydes and 30 to 76 percent of DBP precursors, as seen in Table 1. These levels of control were not found in the pilot-scale biological sand filter having 10 minutes contact time and temperatures ranging from 17° to 27 °C. Table 1 shows for smaller molecules like aldehydes the bench and pilot scale systems were comparable, but for more complex molecules like DBP precursors poorer control was observed under the pilot-scale real world conditions.

Table 1.
Control of Drinking Water Parameters by
Biologically Active Sand

Parameter	Percent Removal[a]	
	Bench-Scale Bioreactors[e,f]	Pilot Plant Filter 5[b,c,g]
Methyl Glyoxal	99,99	97 +/- 2
Formaldehyde	96,d	88 +/- 7
AOC-NOX	d	47 +/- 14
DOC	8,31	16 +/- 9
THMFP	36,20	17 +/- 5
CHCl₃FP	58,32	25 +/- 5
HAAFP	65,68	37 +/- 6
DCAAFP	76,51	41 +/- 6
TOXFP	43,39	26 +/- 7

a O_3/DOC approximately 0.7 mg/mg
b   after steady-state control established
c   based on 163 days of operation
d   no data
e   based on Miltner, Shukairy and Summers (1992)
f   based on Shukairy, Summers and Miltner (1992b)
g   based on Miltner and summers (1992)

SUMMARY
        Ozonation of organic matter leads to the formation of
more biodegradable compounds and to ozonation DBPs. In the
presence of bromide, ozonation also leads to significant
formation of bromate. For a source water that has a high
bromide concentration, the use of ozonation must be
carefully considered because of the high bromate
concentrations that could be generated.
        Aldehydes, ozonation DBPs, are very biodegradable and
can be controlled by biofiltration at both bench and pilot-
scales.
        Ozonation and biotreatment provide good control for
halogenated DBP precursors.   There seems to be no
improvement in the control by ozonation above a certain
ozone to DOC ratio. For Ohio River water, the ratio is
between 0.7 and 1.0 mg/mg. For the individual THM and HAA
species, biotreatment is less effective for the control of
the bromo-substituted compounds because of the increase in
bromide to DOC ratio and   this effect is even more
pronounced at lower chlorine doses.
        The batch-scale bioreactors using sand, with 5 days
residence time, represent ultimate biodegradation. Similar
control   by   biotreatment   was   attainable   for   easily
biodegradable aldehydes in pilot-scale sand filters where
residence times are in the order of minutes. However, for

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the more complex compounds like DBP precursors, poorer control was observed in pilot-scale biological filters than in bench scale bioreactors.

On a pilot-scale, biodegradation of aldehydes was established in a matter of days, AOC-NOX in about a month and DBP precursors in 1.5 to 2.5 months.

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