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BENCH SCALE TREATABILITY OF CONTAMINATED GROUNDWATER AT THE OTT/STORY SITE — PART 1

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Summary

This is the first of a two-part paper describing experimental studies on the treatability of contaminated groundwater at the Ott/Story site in Muskegon, Michigan. This groundwater is severely contaminated by numerous organic compounds. Activated carbon and resin adsorption, aerobic and anaerobic biological treatment, chemical oxidation, and stripping are being investigated at the bench scale. Most treatment technologies studied to date have been moderately effective in reducing the levels of organic contamination. However, a process train consisting of granular activated carbon adsorption followed by activated sludge treatment can achieve high levels of treatment for short periods of time.

Introduction

This paper describes portions of a project being conducted for the U.S. Environmental Protection Agency to evaluate and verify several selected techniques for concentrating hazardous constituents of aqueous waste streams. As reported previously [1,2], these objectives are being met through a multi-phased program involving literature review, desk-top evaluations, and laboratory bench scale treatability studies.

On the basis of an extensive literature review and desk-top analysis, the following unit concentration processes were identified as having potential broad application to the aqueous contamination problems associated with waste disposal sites:

- Biological treatment
- Carbon adsorption
- Chemical coagulation
- Membrane processes
- Resin adsorption
- Stripping.

Although not a concentration technology, because of its demonstrated ability to enhance treatability of numerous organic compounds, chemical oxidation (i.e., ozonation, possibly with UV irradiation) also was judged to have potential application. All of these processes, however, must be supplemented with ancillary processes such as sedimentation and filtration.

Following the desk-top technology evaluations, experimental treatability studies were initiated. The Ott/Story Chemical Company site (now owned by Cordova Chemical Company) in North Muskegon, Michigan was selected for conducting the treatability studies for the following reasons:

- Quantitative data describing the nature and magnitude of the problem was available
- There was no pending litigation which would limit information transfer
- A spirit of co-operation existed between current site owners and the regulatory agencies
- The Michigan Department of Natural Resources was undertaking a feasibility study to identify and evaluate potential clean-up techniques

Groundwater in the area had been contaminated by the disposal and poorly controlled storage of chemical production wastewaters by previous facility owners. The characteristics of groundwater at the Ott/Story site are as follows:

(1) Conventional pollutants

pH	8—12
TOC	400—1500 mg/l

(2) Major priority pollutants

Vinyl chloride	ND—32,500 μg/l
Methylene chloride	<5—6,570 μg/l
1,1-Dichloroethylene	60—19,850 μg/l
1,1-Dichloroethane	<5—14,280 μg/l
1,2-Dichloroethane	350—111,000 μg/l
Benzene	ND—7,370 μg/l
1,1,2,2-Tetrachloroethane	<5—1,590 μg/l
Toluene	<5—5,850 μg/l

In addition to these major pollutants, more than 70 other organic compounds have been detected at concentrations >10 μg/l. Organic compounds are the primary pollutants; heavy metals are believed not to be a problem.

In designing the bench scale studies, it was decided to first examine unit processes and subsequently, as a data base was developed, to evaluate process trains. The bench scale evaluation approach is outlined below:

(1) Pretreatment investigations
   A. Neutralization.
   B. Chemical coagulation and precipitation.
   C. Solids/liquid separation.
   D. Air sparging.

(2) Batch studies
   A. Carbon adsorption.
   B. Resin adsorption.
   C. Chemical oxidation-ozonation.

(3) Batch sequential studies
   A. Air sparging followed by carbon adsorption.
   B. Carbon adsorption followed by air sparging.
   C. Carbon adsorption followed by resin adsorption.

(4) Continuous flow studies — individual processes
  A. Steam stripping, packed bed column.
  B. Granular activated carbon columns.
  C. Biological treatment,
    1. activated sludge.
    2. upflow anaerobic filter.
  D. Biophysical treatment.
(5) Continuous flow studies — process trains
  A. Adsorption/activated sludge.
  B. Carbon adsorption/anaerobic filter.
  C. Carbon adsorption/trickling filter.
  D. Carbon adsorption/anaerobic filter/activated sludge.
  E. Activated sludge/carbon adsorption.
  F. Ozonation/activated sludge.
  G. Ozonation/carbon adsorption/activated sludge.
  H. Ozonation/activated sludge/carbon adsorption.

Bench scale studies through 5B of the above outline are discussed herein. The remaining studies will be discussed in Part 2 of this paper.

Batch studies

Results of pretreatment investigations, batch studies, and batch sequential studies have been reported previously [3]. Major priority pollutant removals resulting from batch studies are summarized in Table 1. To briefly summarize:

- Carbon adsorption reduced almost all organic priority pollutants to less than GC/MS detection limits selected for this study (10 μg/l).
- Resin sorption proved to be only slightly less effective than carbon sorption. Most organic priority pollutants were reduced to below detection limits; all were reduced by at least 98%.
- All volatile organic priority pollutants were reduced to less than detection levels by air stripping. Removals for other organic priority pollutants ranged from 4—96%.
- Carbon treatment of the air sparged groundwater generally resulted in reduction to less than detection limits for the remaining organic priority pollutants. All were reduced by more than 98%.
- Despite good removals of priority pollutants, a significant residual TOC (301—455 mg/l) was measured in all treated samples. This residual represents unidentified non-priority organic pollutants.

Batch groundwater ozonation studies were conducted under the following conditions using a Welsbach Model T-408 laboratory scale ozone generator:

- Ozone production using compressed air.
- Ozone gas flow rate — 2 l/min.
- Ozone dose — approximately 2 g/h (generator operating at 90v).
- Contact time — 2.5 h

TABLE 1

TOC and major organic pollutant removals during batch studies

Compound	Study 1 (mg/l)			Study 2 (mg/l)		
	Raw waste	Resin sorption effluent	Carbon sorption effluent	Raw waste	Air sparge effluent	Sparge plus carbon sorption effluent
TOC	638	455	332	720	641	301
Benzene*	7.8	0.17	0.01	5.3	ND	ND
Benzoic acid	0.17	ND	0.18	0.30	0.02	ND
Camphor	4.0	0.04	ND	3.9	0.47	0.01
Chloroform*	1.4	ND	ND	2.0	ND	ND
1,1-Dichloroethane*	1.2	ND	ND	1.6	ND	ND
1,2-Dichloroethane*	111	0.23	0.01	14	ND	0.01
1,1-Dichloroethylene*	0.06	ND	ND	1.0	ND	ND
Dimethylaniline	17.0	0.25	ND	15.0	0.61	0.08
Ethylaniline	3.3	ND	ND	3.8	0.60	ND
Methylene chloride*	0.06	ND	ND	0.07	ND	NA
Toluene*	2.6	ND	ND	3.6	ND	ND

(*Note*: 27 organic pollutants detected at 0.01 to 0.31 mg/l; none found in effluents.)
ND — Not Detected at detection limit of 0.010 mg/l.
NA — Not analyzed
* — Priority pollutant

- Reactor — glass 18 l vessel fitted with two fritted glass diffusers for mixing and ozone diffusion.
- Batch volume — 7.5 l.

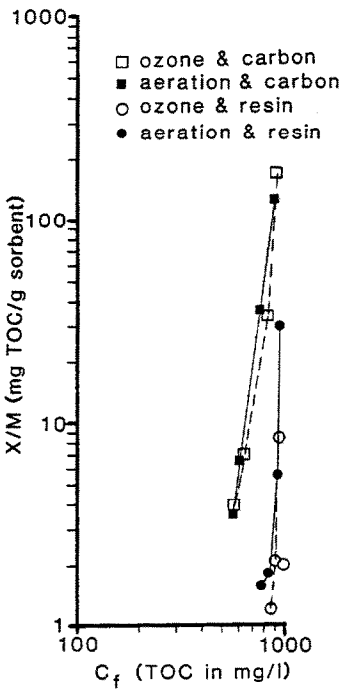
Adsorption isotherms then were developed for activated carbon and resins using ozonated groundwater. To measure the effect of stripping during mixing, an air stripping system was operated in parallel to the ozone system and adsorption isotherms also were developed for the air sparged groundwater.

Batch ozonation effected negligible TOC reduction (comparable to concurrent parallel air sparging). Moreover, ozonation of the groundwater did not significantly enhance TOC removal by activated carbon or resins. TOC removals varied from 14—45% at carbon doses of 0.5—106 g/l and 6—30% at comparable resin doses. Figure 1 shows adsorption isotherms for samples either ozonated or aerated prior to batch contact with activated carbon and polymeric resin. Isotherms resulting from these pretreatment techniques were almost identical and similar to those developed using raw groundwater (3).

Continuous flow studies — individual processes

Steam stripping

Continuous flow, steam stripping experiments were conducted using a



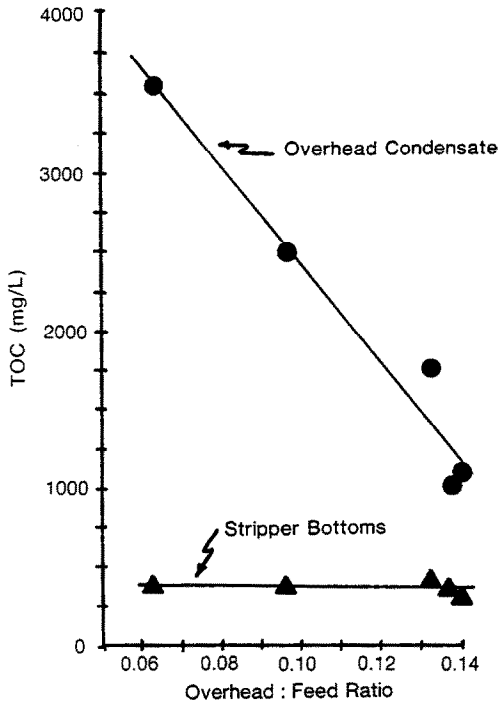


Fig. 1. Adsorption isotherms — effect of ozone and aeration pretreatment.

Fig. 2. Results of steam-stripping studies.

76.2 cm by 4.8 cm (ID) column packed with 6 mm ceramic berl saddles. The apparatus was operated at feed stream flow rates of 40 to 80 ml/min, overhead (condensate) flow rates of 3.5 to 9.2 ml/min (overhead to feed flow ratios of 0.064 to 0.14), no reflux flow, feed stream TOC concentrations of 480 to 610 mg/l, and durations of 1 to 4 hours after establishing steady-state operation within the available operational controls.

As Fig. 2 illustrates, TOC concentration in the stripper bottoms ranged from 300 to 400 mg/l and appeared to be independent of the overhead: feed ratio. Average TOC reduction between feed and bottoms was 34%. These results indicate a major constraint associated with steam stripping — it is necessary to further treat a bottoms waste stream having a flow only slightly less than the feed flow. It should be noted that maintaining steady-state operation of the apparatus proved to be very difficult. The minimum overhead to feed flow ratio which could be achieved was 0.064, significantly higher than the desirable range of 0.02 to 0.05. Thus, stream stripping proved to be only moderately effective and additional treatment of the bottoms would be necessary. Moreover, a method of disposal of the overhead stream would have to be devised. It was concluded that this approach did not have good potential for the Ott/Story problem.

Activated carbon and resin adsorption

Continuous flow granular activated carbon (GAC) studies were conducted at the unadjusted groundwater pH (9.3—10.0) using Calgon* Filtrasorb 300 GAC. Three or four glass columns were arranged in series with sampling ports located at the influent and effluent ends of each column. Each column was 122 cm by 2.54 cm (ID) with 91.4 cm of GAC per column. The system was operated in a downflow mode at a loading rate of approximately 1.35 $l/m^2 s$ (2 gpm/ft^2). This provided an empty bed contact time (EBCT) of approximately 15 minutes per column. Influent TOC concentration varied substantially, ranging from 316—950 mg/l.

Results of a typical study are illustrated in Fig. 3. Results are reported as removal percentage rather than effluent concentration because of influent TOC fluctuations. Generally, after only 3—10 bed volumes (BVs), TOC removal decreased to 50%. This rapid breakthrough was foretold by the steeply sloping isotherms developed in batch tests. TOC leakage reached 90% after about 200 to 240 BVs were processed. Other tests indicated that removal remained at this level until up to 500 BV had been processed.

Continuous flow resin adsorption studies were conducted using Rohm and Haas* XE-347 carbonaceous resin. Three columns similar to those used for GAC studies were charged with 792 to 835 cm^3 of resin and were operated at loading rates of 2.95 to 3.79 BV/h. EBCT ranged from 16—20 min. TOC removal data for one representative study are shown in Fig. 3.

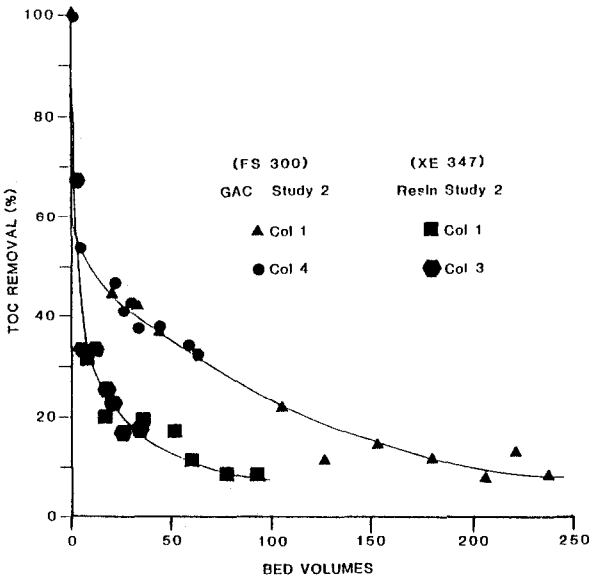


Fig. 3. TOC removal by adsorption.

*Use of trade names does not constitute an endorsement of the product.

As would be expected on the basis of the isotherm data, breakthrough characteristics were similar to those of GAC except that XE-347 TOC removal declined more rapidly. TOC removal diminished to <50% after about 5 BVs were processed and appeared to be stabilized at about 10% for up to 120 BVs loaded.

Biological and biophysical treatment

Several attempts were made to acclimate an activated sludge culture to raw groundwater using 350 ml reactors. All attempts, however, were minimally successful. Neither a conventional activated sludge nor a commercial microbial culture could be acclimated. Slight loading fluctuations encouraged growth of a light colored, filamentous biomass which settled poorly. As shown in Fig. 4, about 40—60% TOC reduction was achieved. However, stripping due to aeration appeared to account for about two-thirds of this removal. Addition of trace elements and nutrients, and pH adjustment to pH 7.0—7.5 did not aid acclimation to raw groundwater. Addition of powdered activated carbon (PAC) at aeration chamber concentrations of about 10,000 mg/l also did not aid acclimation to raw groundwater or improve TOC removal.

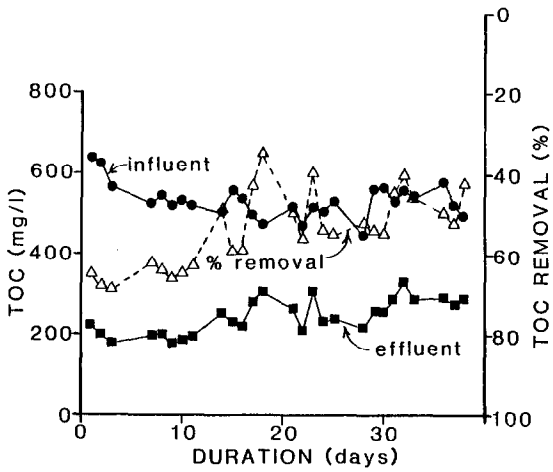


Fig. 4. Acclimated activated sludge treatment of raw groundwater.

Anaerobic biological treatment of pH adjusted, nutrient supplemented groundwater also was attempted. An upflow anaerobic filter (UAF) consisting of a 70.5 cm by 4.76 cm ID glass column packed with ceramic berl saddles was operated at the following conditions:

UAF EBCT — 13.5 h.

UAF average organic loading rate — 1.65 kg TOC/m^3 d (103 lb TOC/1000 ft^3/d)

UAF temperature — 35°C

UAF feed pH — 6.7 to 8.15

As discussed later in this report, the UAF process previously had been a component in a granular carbon adsorption/UAF process train. Consequently, the anaerobic biomass had been acclimated to GAC pretreated groundwater prior to introducing raw groundwater. Within one day after GAC pretreatment was terminated, UAF process performance declined markedly. After about four days, TOC removal by the UAF was <5%, gas production almost ceased, and the ratio of UAF volatile acids to total alkalinity indicated extreme process upset. Without GAC pretreatment anaerobic biological treatment could not be sustained.

Continuous flow studies — process trains

Adsorption/activated sludge

Because of the apparent toxicity of the groundwater to biological treatment systems and the rapid breakthrough of TOC in adsorption systems, it was decided to investigate a combined sorption/activated sludge process train. The rationale was to use the sorbent to protect the biological system from toxic materials. Thus, the sorbent could be allowed to leak relatively high concentrations of organics which would be degraded in the subsequent biological process. Two such process trains were investigated:

(1) Granular activated carbon followed by activated sludge (GAC/AS).
(2) Resin followed by activated sludge (RES/AS).

Operating conditions for the GAC/AS process trains are summarized below:

- GAC — three 91.4 cm columns in series, 0.0815 $m^3/m^2/d$ (2 gpm/ft^2) hydraulic loading, and 34 min EBCT.
- AS — one liter reactors with hydraulic retention times (HRT) of 6—16 h; GAC effluent neutralized and nutrients added; mixed liquor suspended solids ranged from 2000 to 8000 mg/l.

Performance of this process train under various conditions is illustrated in Fig. 5. The GAC process was operated approximately 6 h/day, five days per week whereas the two AS reactors were operated continuously. This necessitated collection and storage of GAC effluent for 1—3 days to maintain continuous feed to the AS reactors. Some decrease in TOC levels were noted during the storage periods. Losses probably were due to volatilization or biological degradation. Thus, it is assumed that similar reductions would have occurred in the AS reactors. The following observations have been made on the basis of data presented in Fig. 5:

- GAC pretreatment of raw groundwater permits development of a culture of aerobic organisms capable of further treating GAC effluent. In excess of 95% TOC removal can be achieved by this process during the period which GAC removal of TOC exceeds 30%. After this initial period, process train performance declines as GAC performance declines. Effluent TOC could be maintained at <100 mg/l only for short time periods and only when GAC performance was at its peak.

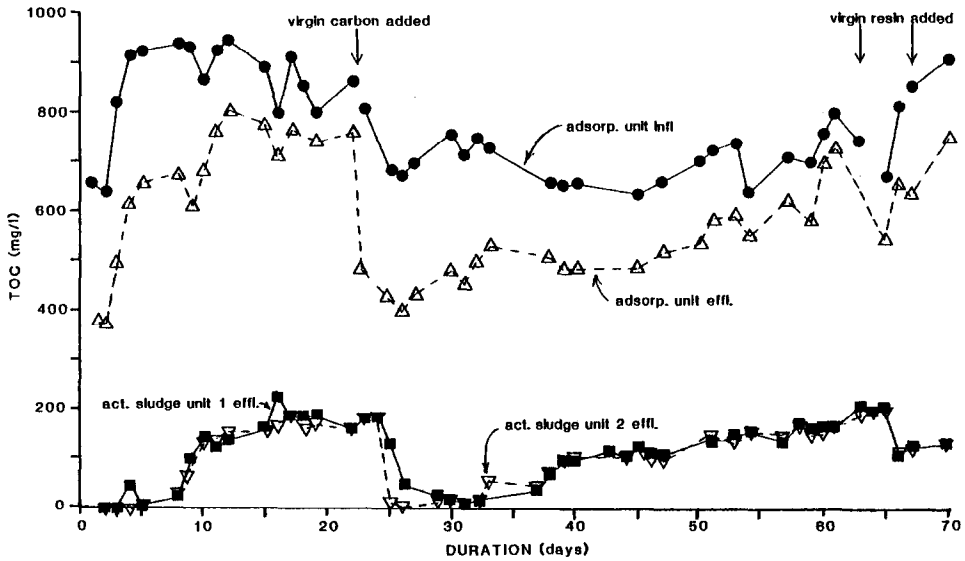


Fig. 5. Performance of adsorption/activated sludge process train.

- Some fraction of TOC which initially is sorbed by GAC begins to leak through the system after a short period of operation. This fraction of TOC which leaks through the GAC system is not toxic to AS but does not appear to be removed or reduced either biologically or by air stripping associated with AS aeration.
- AS process HRT in the 6 to 16 h range had little impact on process performance (based upon TOC removal).
- Overall system performance was maintained at 75—85% TOC removal (effluent TOC of 100—185 mg/l) for about 21 days (>110 BVs processed by GAC).
- Visual observations and typical mixed liquor analyses (MLSS and MLVSS) suggest that the biological systems could survive in and utilize GAC pretreated groundwater even after GAC performance had declined to about 10% TOC removal. However, AS effluent contained about 200 mg/l TOC at this time.

As shown in Table 2, high levels of organic priority pollutant removals can be attained by the GAC/AS train even with effluent TOC concentrations of 100—200 mg/l. Almost all of the organic priority pollutants detected in raw groundwater were removed consistently to less than the level of detection (0.01 mg/l) by the process train. One consistent feature of these data and previous GC/MS analyses from batch carbon adsorption studies is the early leakage of 1,2-dichloroethane. A few other compounds (benzene, methylene chloride. and toluene) also were detected to have broken through the carbon in some batch and continuous flow studies. Acid and base—neutral extractable compounds generally did not break

TABLE 2

Granular activated carbon/activated sludge process train performance (mg/l)

Compound	Collected on Day 2*		Collected on Days 9 and 10*			Collected on Day 17*	
	Raw groundwater	GAC effluent	Raw groundwater	GAC effluent	AS effluent	GAC effluent	AS effluent
TOC	637	380	929	604	90	770	183
Total cyanide	NA	NA	0.11	0.21	0.23	0.23	0.20
CN_A	NA	NA	<0.05	<0.05	<0.05	<0.05	<0.05
Total phenol	NA	NA	16	<0.16	<0.10	<0.10	<0.10
Methylene chloride	2.1	0.029	14	0.01	ND	0.16	ND
1,1-Dichloroethene	1.6	ND	0.06	0.01	ND	ND	ND
1,1-Dichloroethane	2.4	ND	0.17	0.02	ND	ND	ND
Trans-1,2-dichloroethane	0.06	ND	0.04	ND	ND	ND	ND
Chloroform	9.8	ND	0.07	0.06	ND	ND	ND
1,2-Dichloroethane	72	ND	25	1.4	ND	0.05	ND
1,1,1-Trichloroethane	7.6	ND	0.39	0.04	ND	ND	ND
Trichloroethylene	0.06	ND	0.03	ND	ND	ND	ND
Benzene	1.2	ND	1.5	0.02	ND	ND	ND
1,1,2-Trichloroethane	0.11	ND	0.07	ND	ND	ND	ND
Perchloroethylene	0.49	ND	1.9	ND	ND	ND	ND
Toluene	2.3	ND	0.97	0.05	ND	0.01	ND
Chlorobenzene	0.23	ND	0.29	ND	ND	ND	ND
Phenol	0.025	ND	0.028	ND	ND	ND	ND
2-Chlorophenol	0.040	ND	0.036	ND	ND	ND	ND
2,4-Dichlorophenol	0.010	ND	0.010	ND	ND	ND	ND
1,2-Dichlorobenzene	0.085	ND	0.077	ND	ND	ND	ND
Dibutyl phthalate	ND	ND	ND	ND	0.05	ND	ND

NA — Not analyzed
ND — Not detected
No other priority pollutants detected at 0.01 mg/l detection limit
*— Refers to Fig. 5.

through the GAC process. Data in Table 2 also indicate that the activated sludge process completely removed the few organic priority pollutants leaking through the GAC system even though TOC removal declined. The continued removal of organic priority pollutants may be due to stripping.

A limited effort was made to determine if specific organic priority pollutants were concentrated in the biological sludge. Analysis of a single sludge sample indicated the absence of organic priority pollutants at a 0.01 mg/l detection level.

An off-gas sample from the aerated reactor was collected using a coal trap (acetone and dry ice) to condense off-gas vapors. Air flow to the reactor was approximately 2 l/min and the collection period was 4 hours. The following organic priority pollutants were detected in this sample:

Methylene chloride	1.02 μg/l air
1,2-Dichloroethane	1.04 μg/l air
Benzene	0.25 μg/l air
Perchloroethylene	0.125 μg/l air
Toluene	0.088 μg/l air

As indicated in Fig. 5, on day 64, the pretreatment process was changed from GAC adsorption to resin adsorption (carbonaceous resin XE-347). The operating conditions for two resin-adsorption pretreatment studies were as follows:

- 3 columns in series.
- Columns were 2.54 cm diameter.
- Total BV = 792—835 cm^3.
- Downflow operation at 41—50 ml/min (2.95—3.79 BV/h).
- EBCT ranged from 16—20 min.

As shown in Fig. 5, the AS units loaded with resin-treated groundwater were not able to reduce TOC levels to <100 mg/l even for a short period after virgin resin was placed on line. AS performance at 4 h and 8 h HRTs did not differ.

Following investigation of pretreatment with XE-347 resin, GAC pretreatment was resumed. The process train was operated for several more cycles of carbon loading and replacement with similar results. That is, short periods of greater than 95% TOC removal followed by gradual decline as GAC removal decreases.

Adsorption/anaerobic biological treatment

With the thought of minimizing potential air pollution due to stripping of volatile organics during biological treatment, an attached-growth upflow anaerobic filter was investigated. The process was preceded by GAC pretreatment. Flow through the GAC unit was reduced from previous studies to facilitate continuous operation of the two unit processes (direct feed from the GAC unit to the UAF). Operating conditions for the process train are as follows:

Flow:	1.15—2.0 ml/min, 1.71 ml/min average
GAC column:	78.7 cm × 2.54 cm ID glass
GAC EBCT:	3.3—5.8 h, 3.9 h average
UAF column:	88.6 cm × 4.76 cm ID glass with 70.5 cm of packing
UAF EBCT:	13.1--22.8 h, 15.4 h average

The UAF reactor was filled with ceramic berl saddles and then with sludge from a well operated municipal wastewater sludge anaerobic digester. The UAF reactor initially was fed raw sewage for eight days prior to converting to GAC pretreated groundwater.

Performance data on the initial month of operation of the GAC/UAF process train on pretreated groundwater are shown in Fig. 6. TOC removals by the various steps in the process train are summarized below:

	Removal	
	Average	Range
GAC/UAF train	66%	31—81%
GAC process	31%	10—46%
UAF process	50%	12—67%

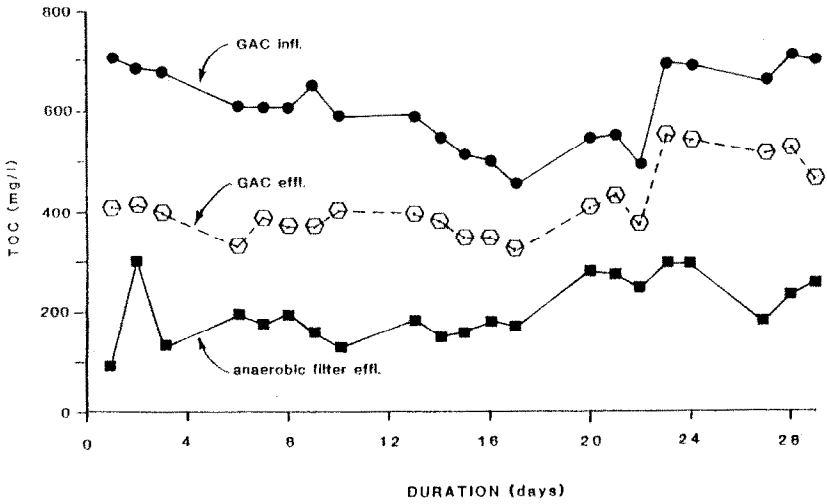


Fig. 6. Performance of GAC/anaerobic filter process train.

Gas production during this month averaged 505 ml/g TOC fed. Figure 6 indicates that UAF effluent TOC increased as TOC leakage from the GAC pretreatment process increased. However, TOC removal and gas production data cannot be related to organic loading rate or other operational data such as sludge pH, sludge total alkalinity, or volatile acids production. In an attempt to bring the pH into a range reported to be most optimal (pH 7.2—7.6), the GAC influent pH was adjusted to about 7.0—7.5. This had no apparent effect on performance.

Summary

Laboratory experiments on contaminated groundwater at the Ott/Story Chemical Company site in Muskegon, Michigan were conducted. Results indicate good removals of volatile priority pollutants by batch air stripping. In addition, batch carbon and resin sorption reduce all organic priority pollutants by greater than 98%. However, high effluent TOC levels have been measured subsequent to all applied treatments. This TOC represents organic contamination by non-priority pollutants.

Steam stripping in a packed column resulted in concentration of TOC in the overhead condensate stream. However, within the range of feed flow and overhead flow rates investigated, TOC of the bottoms could not be reduced below about 400 mg/l. Overhead TOC approached 4,000 mg/l at an overhead rate of approximately 6% of the feed flow.

Granular activated carbon (GAC) employed in continuous flow small size columns was not capable of sustaining high levels of TOC removal. TOC removal declined to <50% after processing <5 BV. Within 100—160 BV loaded, TOC removal declined to 10 to 15% and remained at this level for

up to 200 BV. GAC adsorption was capable of achieving high levels of organic priority pollutant removals even when TOC removal had declined to 35% and effluent TOC levels were ≅ 600 mg/l. In both batch and continuous flow adsorption studies, some volatile priority pollutants were detected in the effluent. None of the acid or base—neutral extractable organic priority pollutants detected in the raw groundwater were found in GAC effluent after processing up to 75 BV.

In continuous flow small size column studies, carbonaceous resin demonstrated TOC breakthrough characteristics similar to those of GAC. However, TOC breakthrough occurred more rapidly.

GAC pretreatment of raw groundwater permits development of a culture of aerobic organisms capable of further treating GAC effluent. In excess of 95% TOC removal can be achieved by this process during the period which GAC removal of TOC exceeds 30%. After this initial period, process train performance declines as GAC performance declines. Several organic priority pollutants were detected in off-gas from AS reactors; these included methylene chloride, 1,2-dichloroethane, benzene, tetrachloroethylene, and toluene. No organic priority pollutants were detected (at a detection limit of 10 μg/l) in an AS biomass sample.

Anaerobic treatment (upflow anaerobic filter, UAF) of GAC pretreated groundwater is possible. However, UAF performance declines as GAC performance declines (although changes in UAF organic loading rate do not appear to effect UAF performance). Overall, the GAC/UAF process train performs more poorly than the GAC/AS process train with an upper TOC removal limit of 81%.

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