Evaluation of an ozonation system for reduced waste sludge generation

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Abstract The ultimate disposal of biosolids has been and continues to be one of the most expensive problems faced by wastewater utilities. Previous work has shown that the waste sludge generation in an activated sludge plant can be reduced by promoting cryptic growth conditions (i.e., biomass growth on intracellular products). For this purpose, excess biosolids from a continuous flow activated sludge system were solubilized using ozone as the cell lysing agent, and then returned to the aeration tank. The results of these preliminary studies indicate that the proposed process configuration has the potential to reduce the waste sludge production by 40% to 60%. In the present research, the details of the ozonation process is further investigated to determine the maximum solubilization efficiency. For this purpose, a number of variables such as the solids concentration in the excess sludge, ozonation time, and ozonation dosage rate are studied.

Keywords Activated sludge; biosolids management; cryptic growth; microbial yield; ozonation

Introduction

In a typical municipal wastewater treatment plant, the handling, treatment, and ultimate disposal of wasted biosolids is one of the major problems, accounting for 50 to 60% of the operating costs of the plant. This problem is expected to increase with population growth and tightening of regulations on biosolids disposal. While the ideal solution to biosolids management is to develop a sludgeless process, a more realistic and tenable solution would be to minimize the sludge generation in the first place. Previous laboratory studies have demonstrated that the net biomass growth could be reduced under cryptic conditions (Hamer, 1985; Mason and Hamer, 1987; Canales *et al.*, 1994; Yasui and Shibata, 1994).

Based on these findings, it was hypothesized that cryptic growth may be a feasible approach to achieve the goal of reduced sludge production. This hypothesis was tested in continuous flow bench scale activated sludge systems, fed with a synthetic feed. Two activated sludge reactors were run in parallel: one as a control system and one as a test system with recycling of excess sludge that was solubilized by ozonation. The results of this study indicated that the proposed process can reduce the waste sludge production by 40 to 60%. The ozonation dose used in this study was 0.2 mg O_3/mg SS per hour and the ozonation time was chosen as 3 hours. The details of this research are presented elsewhere (Egemen *et al.*, to appear).

In this study, the details of the ozonation process are evaluated using a number of batch scale studies. It is proposed that the ozonation process can be configured to achieve the maximum amount of solubilization with minimum ozone dose. The specific objective of this paper is to determine the most feasible and cost effective ozonation time and oxonation dose. The efficiency of solubilization is evaluated based on the soluble chemical oxygen demand (SCOD) concentration of the wasted sludge samples before and after ozonation.

Materials and methods

Increase in SCOD with ozonation

One set of experiments was performed to demonstrate that SCOD of the sludge samples

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increase as a result of ozonation, and this increase is mainly due to cell lysis and solubilization of the internal cell matter. Excess sludge from the lab scale continuous flow reactors was split equally into two samples. One sample was filtered and the supernatant was ozonated; whereas, the other sludge sample was directly ozonated without filtering. Both sludge samples were ozonated at the same ozone dosage for one hour. SCOD concentrations of the samples were determined at the beginning and at the end of the experiments.

Ozonation of the samples was achieved in an acrylic bubble column of 1.75 in ID. Although the depth of the column varied depending on the amount of sludge ozonated, it was mostly around 2 feet deep. Ozone was introduced into the column through a brass diffuser located on the bottom of the column. The diffuser was made of a 0.125 inch brass tubing with 0.04 inch diameter hole drilled in it.

Ozonation period
These preliminary studies were performed to understand the ozonation process, to determine the approximate time required for ozonation of sludge and to provide feedback for the detailed analysis of ozonation process.

Excess sludge from the lab scale activated sludge reactors was ozonated for different periods of time and with varying ozone dosages. The ozone dose used was varied between 0.10 to 0.43 mg O_3 per suspended solids (SS) per hour. Since the purpose of these experiments was to describe the ozonation and lysing processes assuming ozone was not the limiting agent, ozone was supplied to the system in excess. The amount of ozone leaving the system was not determined; therefore the data is compared based on the amount of ozone input to the system rather than the net amount of ozone transfer. The ozonated sludge samples were analyzed for SCOD concentrations and cell viability as a function of time. Also, the total suspended solids (TSS) concentration of the sludge was determined at the beginning of the experiment. The same bubble column described above was used during these preliminary ozonation studies.

Depth of ozonation column
Batch studies were performed to determine the effects of the depth of the ozonation column on the efficiency of ozonation. In order to carry out the experiments with a more homogeneous and consistent sludge, this set of experiments was performed using the return activated sludge samples taken from Las Cruces, NM, municipal wastewater treatment plant.

The initial SCOD and TSS concentrations of the activated sludge samples were determined at the beginning of each experiment. Based on these measurements, the return activated sludge samples taken from the city wastewater plant were diluted in order to make sure that the sludge ozonated on different days had approximately the same SCOD and TSS concentrations at the beginning of the ozonation process. The diluted activated sludge samples were ozonated for one hour at depths of 2, 3, 4, 5, and 6 feet at a constant ozone dose. The SCOD of the sludge samples were analyzed both at the beginning and at the end of the ozonation period.

These experiments were performed using an external loop airlift reactor of 2 feet. The riser was 0.5 inch diameter tygon tubing, which was connected to a 0.5 inch diameter downcomer that was made of PVC pin. Ozone was introduced into the riser through a valve located on the side of the tygon tubing at 2 feet. The length of both the PVC pipe and tygon tubing was shortened based on the reactor length to be tested.

Determination of ozone dosage and input frequency
Batch studies were performed to evaluate the effect of ozone dosage and ozone input frequency on the degree of cell lysis and solubilization. Excess sludge from the lab scale

continuous flow reactors was ozonated with different ozonation rates (i.e., different gas phase ozone concentrations and different air flowrates) for mostly three hours. The TSS concentrations of the sludge samples were also changed by concentrating the sludge to determine the effect of varying TSS concentrations. The solutions were sampled as a function of time and analyzed for SCOD concentrations and cell viability. The ozone leaving the system was determined using potassium iodine traps described in Method 2350E of *Standard Methods* (APHA, AWWA, WEF, 1995).

Based on the results of depth of ozonation column studies, an internal loop airlift reactor was built. The column consisted of two concentric acrylic tubes; the inner acting as the downcomer and the annular space between the two tubes serving as the riser. The outer tube was of 1.5 inch ID × 27 inch height; and the inner tube of 1 inch ID × 24 inch height. Acrylic guides were used to centre the inner tube within the outer tube and to raise it 0.5 inches off the bottom of the reactor.

The ozone laden air stream was introduced into the riser of the ozonation column through a 0.125 inch OD brass diffuser port, which had one 0.04 inch diameter hole drilled in it. The diameter of the outer column was increased to 3.5 inch at a height of 27 inches in order to be able to hold larger volumes of sludge without significantly increasing the column depth. The top cap of the column was equipped with an air-tight valve to capture off-gases, which were then diverted to the glass washing bottles for determination of excess ozone.

Results and discussion

Effect of ozonation on excess cells

Mechanistically, ozone is proposed to act as an oxidant resulting in the rupture of filaments and the cell wall. The degree of cell lysis depends on the dose and duration of ozone application. The results of this study demonstrate that the SCOD content of the sludge increases and the SS concentration decreases as a result of ozonation. It was necessary to demonstrate that this increase in the SCOD of the excess sludge as a result of ozonation was mainly due to cell lysis and release of intracellular organic matter. A summary of the results of experiments for two different sets of runs is presented in Figure 1. As can be seen from the figure, the SCOD of the filtered solution did not increase with ozonation, whereas that of the unfiltered solution with bacteria in it did. Therefore, it was concluded that ozonation results in cell lysis and the increase in SCOD was mainly due to cell lysis.
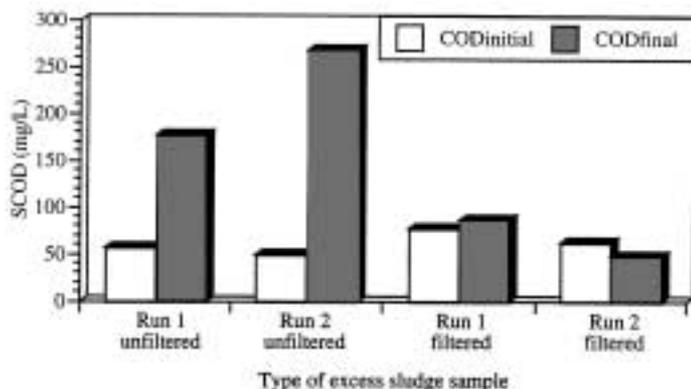

Figure 1 SCOD concentration of filtered and unfiltered excess sludge samples at the beginning (CODinitial) and end of one hour ozonation period (CODfinal)

Ozonation period

A large number of experiments were run in order to determine the average ozonation period to be used in the experiments. Of the large number of batch studies performed, as a typical example case, the SCOD and the decrease in the number of colony forming units (N/N_o) as a result of continuous ozonation in one of the trials are plotted in Figure 2. Similar results were obtained with every run.

As can be seen from Figure 2, SCOD increased as ozonation continued, reached a maximum around three hours, and stayed constant thereafter. The number of colony forming units (N) decreased by about 2 orders of magnitude at the end of three hours. Although N continues to decrease after 3 hours, SCOD remains constant. This implies that after three hours of ozonation, the amount of COD solubilized by ozone is equal to the amount of dissolved organic matter oxidized by ozone, thereby resulting in no net change in the SCOD concentration. Although the ozonation period is closely related to the ozone dosage, ozone gas phase concentration, and TSS concentrations, as will be discussed in later, 3 hours was accepted as the average ozonation time to be used.

During the course of the experiments, it was realized that the SCOD of the solution increases although ozone input is stopped after some ozonation. This was true especially after a certain amount of ozone was already dissolved in solution. After that point (which was usually about an hour), majority of the ozone was lost in the off-gas and not transferred into the solution.

Therefore, in order to determine the effect of ozone input frequency and pattern, another set of experiments was performed by varying the ozonation time and determining the SCOD as a function of time. A summary of results is given in Table 1. Based on these results, the SCOD concentrations with only one hour of ozonation were slightly lower than those with two hours of ozonation.
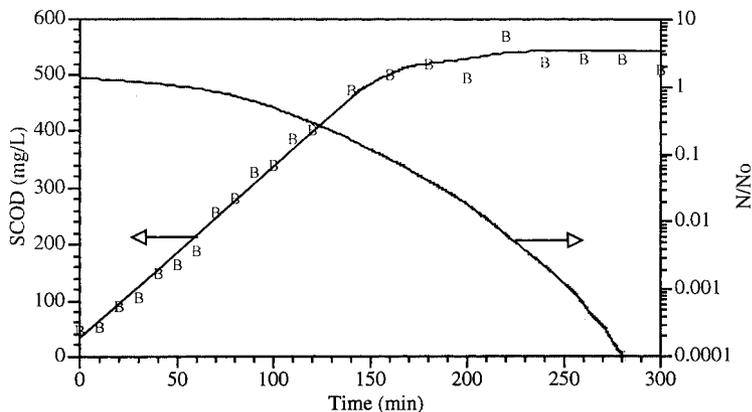

Figure 2 SCOD and *N/No* as ozonation proceeds

Table 1 SCOD measurements with varying ozone input frequency

	SCOD concentrations (mg/L)	
Time (hrs)	One hour ozonation time	Two hour ozonation time
0	50	121
1	268	not determined
2	304	369
3	303	467
4	not determined	448
5	not determined	566
6	not determined	578
18.5	not determined	553

In order to further examine the optimum input frequency, the ozone was introduced into the system only every other 15 min intervals, and completely stopped after two hours. SCOD profile as a function of time under these conditions is given in Figure 3 and a comparison between intermittent and continuous ozonation is provided in Table 2. Based on these results, it was concluded that the mass transfer of ozone into water is the faster reaction of the proceeds, and the reaction of the ozone with the cell material is the slower step.

Reactor configuration

In order to determine an efficient reactor configuration, the solubilization efficiency of ozone was compared using two different reactor configurations: a bubble column versus an airlift reactor. The efficiency of solubilization was defined as the amount of mass of COD increase in the ozonated sludge per mass of ozone input into the system. In this set of experiments, the amount of ozone leaving in the off-gas was not measured; therefore, the data is compared on the basis of ozone input. The variations in SS and gas phase ozone concentrations were unintentional. A summary of mass of COD increase per mass of ozone delivered into the system is given in Table 3 for bubble column and airlift reactor.

Considering the fact that an airlift configuration provided more efficient mixing, the airlift reactor described in the methods section was used for the rest of the experiments.

Depth of ozonation column

A total of 2 batch experiments were performed at different column depths. For each depth tested, triplicate SCOD samples were prepared at the end of one hour ozonation period, resulting in a total of 78 data points. The initial SCOD and MLSS concentrations were 20–50 mg/L and 1000–1100 mg/L, respectively. The mean and standard deviation of the SCOD concentrations at the end of the ozonation period are plotted in Figure 4.
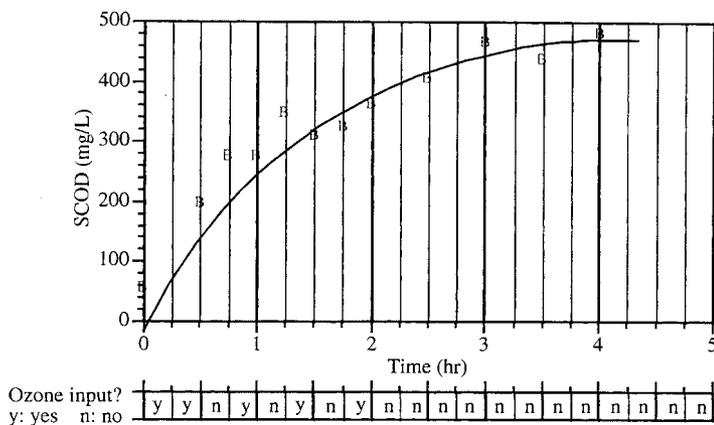

**Figure 3** SCOD generation as a result of intermittent ozonation

Table 2 Comparison between intermittent and continuous operation

Sampling time (hrs)	Actual ozonation time (min)	SCOD at the sampling time (mg/L)
2	75 (intermittent)	363
2	120 (continuous)	369
3	120 (intermittent)	467
3	180 (continuous)	467
4	120 (intermittent)	481
4	240 (continuous)	448

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Table 3 Comparison of bubble column and airlift reactor

Bubble column			Airlift reactor		
Ozone gas conc. (mg/L)	SS conc. (mg/L)	Efficiency*	Ozone gas conc. (mg/L)	SS conc. (mg/L)	Efficiency*
9.5	1300	0.26	6.4	1110	1.25
9.5	1210	0.24	9.2	1660	0.41
9.5	1550	0.13	13.5	1290	1.19

*given as mg COD increased per mg O_3 delivered into the system
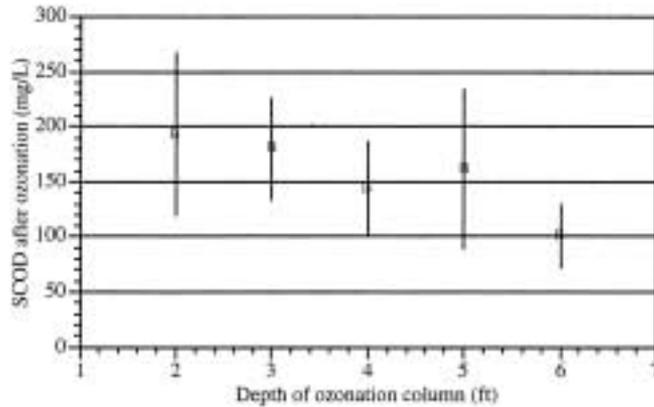
Figure 4 Effect of ozonation column depth on the solubilization

A hypothesis testing analyses was carried out to determine whether the depth of the ozonation column affects the final SCOD, i.e., whether the slope of the regression of SCOD vs depth is zero or not. Based on the statistical analyses, the hypothesis that the slope of regression is zero was accepted at 99% confidence. Therefore, it was concluded that the ozonation column depth did not significantly effect the solubilization efficiency and a 2 ft airlift ozonation column was selected.

Determination of ozonation rate

One of the most important parameters of the proposed process is determination of the ozonation rate required to achieve higher system performance. Ozonation rate was defined as the amount of ozone that must be transferred into the system per hour per mass of SS in the sludge to be ozonated. In order to find out the key parameters of ozonation, another variable, solubilization rate was introduced. Solubilization rate was defined as the mass of SCOD increased as a result of ozonation per a given time.

Based on the results of determination of ozonation period experiments, the sludge was mostly ozonated for three hours, although there were some exceptions to this. The results of these experiments were very similar to those given in Figure 2. The SCOD increase and cell viability studies yielded similarly shaped curves, as was the case in bubble column.

In order to determine the range of ozonation rate that can be applied and to determine the relationship between the different variables of the process, a number of experiments were performed. Based on these experiments, it was observed that the solubilization rate depends not only on the ozonation rate applied, but also, the gas phase ozone concentration as well as the SS concentration of the sludge ozonated. The individual relationships between the gas phase ozone concentration as well as the SS concentration and solubilization rate are plotted in Figures 5 and 6.
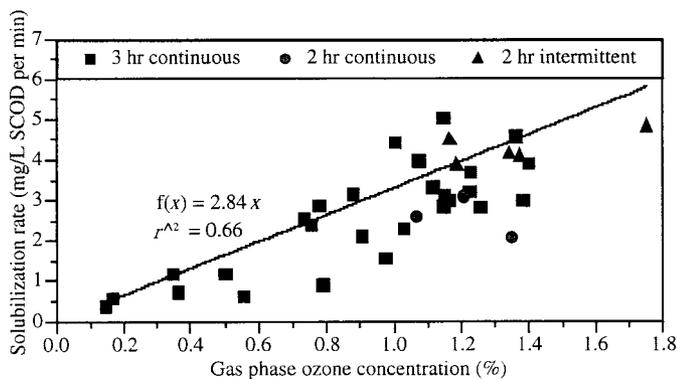
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Figure 5 Solubilization rate as a function of gas phase ozone concentration
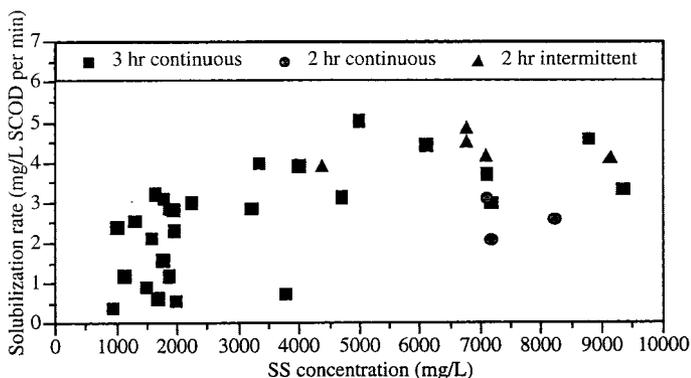

Figure 6 Solubilization rate as a function of SS concentration of the sludge

In order to further examine the relationship between these variables and solubilization rate, a stepwise regression was applied to all the data collected. Three independent variables were studied: ozonation rate, gas phase ozone concentration, and SS concentration. The dependent variable was chosen as the solubilization rate, as determined from the slope of the SCOD line when plotted as a function of ozonation time in minutes (as an example, see the slope of the line given in Figure 2). A stepwise regression performed on the total of 35 data points (including 2 hour continuous ozonation, and intermittent ozonation data) yielded a multiple linear regression using only the gas phase concentration and SS concentration. The equations obtained from different stepwise regressions are summarized in Table 4.

Based on the equations given in Table 4, the following equation was accepted as the design approximation for the ozonation system:

$$2.3\ O_3\ (\%) + 0.14\ SS\ (g/L) = SR\ (mg \cdot SCOD/min)$$

Table 4 Summary of stepwise regressions using the ozonation rate data

Data used	Stepwise regression equation*	Adj r^2
All	2.288 O_3 (%) + 0.144 SS (g/L) –0.02 = SR	0.707
All except intermittent	2.269 O_3 (%) + 0.137 SS (g/L) –0.016 = SR	0.647
Only intermittent	No equation could be generated	

*Sr: Solubilization rate (mg·SCOD/min), O_3: Ozone concentration

The correlation matrix was checked to make sure that the variables of the developed equation are independent of each other and can be used in the approximation of solubilization rate.

It was concluded that the solubilization rate dramatically increased when the gas phase ozone concentration increased. Increase in gas phase ozone concentration increases the mass transfer driving force, and therefore the mass of ozone transferred into the solution also increases. Considering the fact that the field scale ozone generators can generate ozone laden air streams with high concentrations, it is acceptable to design the process requiring high gas phase ozone concentrations.

Similarly, it was concluded that the solubilization rate increases when the SS concentration of the sludge ozonated increases. As can be seen from Figure 6, the solubilization rate stayed practically constant at SS higher than about 300 mg/L. Such concentrations can easily be expected in the field, when the sludge is wasted from the secondary settling basins.

Conclusions

Previous laboratory scale studies indicate that the proposed process can reduce the waste sludge production by 40 to 60%. However, the ozonation process needs to be studied in depth in order to increase the feasibility of the process. Based on the batch studies performed in this research, it was concluded that an average ozonation time of 3 hours per day is acceptable. However, intermittent ozonation is preferred over continuous ozonation due to increased solubilization rates. In addition, it was accepted to be feasible to design the process using high gas phase ozone concentrations, such as 1.5% to 2% and ozonate sludge that has SS concentrations higher than 3000 mg/L.

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