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# Activated Sludge Degradation of Nitrilotriacetic Acid (NTA) - Metal Complexes



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# ACTIVATED SLUDGE DEGRADATION OF NITRILOTRIACETIC ACID (NTA) - METAL COMPLEXES

by

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## ABSTRACT

Bench scale batch activated sludge experiments were conducted to determine the biodegradability of nitrilotriacetic acid (NTA) complexes with calcium (Ca), iron (Fe), lead (Pb), chromium (Cr), copper (Cu), zinc (Zn), cadmium (Cd), nickel (Ni) and mercury (Hg). Degradation rates were evaluated at NTA levels of 8 and 16 mg/L as $H_3NTA$, temperatures of 5° and 15°C, and various metal concentrations.

It was determined that the NTA complexes with Ca, Fe, Al, Pb, Cr, Cu and Zn at temperatures of 10° to 15°C degraded readily, with first-order degradation coefficients in the range of -0.05 to -0.10 $hr^{-1}$. The heavy metal NTA complexes of Cd and Ni and Hg degraded poorly, with degradation coefficients of less than -0.02 $hr^{-1}$. With the exception of NTA complexes with Zn, all degradation rates were reduced considerably at 5°C.

The apparent degradation coefficient of zero for mercury is suspected to be due to a toxic effect on the activated sludge rather than to the formation of a bio-resistant complex.

These experiments, reinforced by observations of other related studies, led to the conclusion that buildup of NTA in the aquatic environment even during low winter temperatures is extremely unlikely.

# RÉSUMÉ

Afin de déterminer la biodégradabilité de l'acide nitrilotriacétique (NTA) combiné au calcium (Ca), au fer (Fe), au plomb (Pb), au chrome (Cr), au cuivre (Cu), au zinc (Zn), au cadmium (Cd), au nickel (Ni) et au mercure (Hg), on a mené des expériences en laboratoire avec des boues activées. Les concentrations de $H_3NTA$ étaient de 8 et de 16 mg/L, les températures de 5 et 15°C, et les métaux avaient diverses concentrations.

Entre 10 et 15°C, les complexes de Ca, Fe, Al, Pb, Cr, Cu et Zn se dégradaient rapidement, selon un coefficient d'ordre un, dans un intervalle de -0.05 à -0.10 $h^{-1}$. Les complexes de Cd, Ni et Hg se sont faiblement dégradés (coefficients inférieurs à -0.02 $h^{-1}$). Exception faite du complexe de Zn, tous les autres se sont dégradés beaucoup moins rapidement à 5°C.

Le coefficient apparemment nul de la dégradation du complexe mercurique résulterait d'un effet toxique exercé sur les boues activées plutôt que de sa résistance aux microorganismes.

Ces expériences, renforcées par des observations faites au cours d'études analogues, permettent de conclure qu'il est fort peu probable que le NTA s'accumule dans le milieu aquatique, même aux basses températures d'hiver.

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# CONCLUSIONS

Ninety bench scale batch activated sludge experiments were conducted on NTA-metal complexes at initial NTA levels of 8 and 16 mg/L as $H_3NTA$, temperatures of 5° and 15°C, and various metal concentrations. On the basis of the study results, the following conclusions have been drawn:

1. The NTA-metal complexes could be classified into two groups:
   (i) a readily degradable group consisting of the metals calcium, iron, aluminum, lead, chromium, copper and zinc with first-order degradation coefficients at 10° to 15°C ranging from -0.05 to -0.10 $hr^{-1}$; and
   (ii) a degradation resistant group consisting of the metals cadmium and nickel having degradation coefficients less than -0.02 $hr^{-1}$.
2. NTA-metal complex degradability was not significantly affected over a wastewater temperature range of 10° to 15°C; however, degradability was significantly reduced at 5°C.
3. Interference of NTA with alum, iron and lime phosphorus removal systems is expected to be insignificant.
4. Since concentrations of cadmium, nickel and mercury found in domestic wastewaters are usually low, poor degradability of these NTA-metal complexes is a problem of little practical significance.
5. The data collected from these experiments and other related study data indicated that a buildup of NTA in the aquatic environment, even during low winter temperatures, is extremely unlikely.

# 1 INTRODUCTION AND STUDY OBJECTIVES

Considerable research has been devoted to determining the environmental implications of using trisodium nitrilotriacetate (NTA) as a laundry detergent builder. Epstein (1972), Thom (1971), Thayer and Kensler (1973) and Prakash (1976) have compiled extensive reviews on this subject. The biodegradability of NTA when subjected to conventional wastewater treatment processes has been examined by Eden et al (1972). Degradation of NTA in receiving streams has been reported by Swisher et al (1973) and Shannon et al (1974). These studies have demonstrated that, at wastewater and water temperatures of less than 10°C, NTA degradation rates are reduced. In addition, previous research has shown that NTA is capable of forming very stable soluble complexes with certain metals such as nickel, cadmium and mercury (Chau and Shiomi, 1972; Gudernatsch, 1970). These complexes are, in turn, quite resistant to biodegradation, thereby presenting the potential for increasing metal transport through conventional waste treatment facilities. Data presented by Walker (1974) reveals that the biodegradation of cadmium, nickel and copper complexes is improved somewhat in the presence of iron or water hardness.

Evidence has been presented by Manning and Ramamoorthy (1972) that mixed ligand (NTA-metal-phosphate) complexes can be formed which are extremely stable in neutral or alkaline solutions. Their experiments carried out at laboratory scale conditions in pure solutions, suggested that the existence of these mixed ligand complexes in wastewaters could reduce the efficiency of phosphorus removal systems which use iron or aluminum salts as the precipitant.

In August, 1970 Canada passed legislation restricting the phosphorus content in laundry detergent formulations to no more than 20%, as $P_2O_5$, with a further reduction to a maximum of 5%, as $P_2O_5$, effective January 1, 1973. As a result, the use of NTA as a builder replacement for phosphates has increased. A considerable amount of research has been conducted in Canada on the environmental implications of NTA usage. A large part of this research as it pertains to wastewater treatment has been carried out by the Environmental Protection Service at the Wastewater Technology Centre in Burlington.

This report describes the results of a bench scale program designed to determine the rate and degree of degradation of various NTA-metal complexes when subjected to activated sludge treatment. The effect of temperature upon the biodegradation of these complexes was also investigated.

# 2 EXPERIMENTAL PROCEDURES

## 2.1 NTA-Metal Complexes

Mixed ligand complexes of the form NTA-metal-phosphate were prepared in 1:1:1 molar ratios. Stock solutions of these complexes were made up for the following metals: $Al^{3+}$, $Cd^{2+}$, $Ca^{2+}$, $Cr^{3+}$, $Cu^{2+}$, $Fe^{3+}$, $Hg^{2+}$, $Ni^{2+}$, $Pb^{2+}$ and $Zn^{2+}$. The source salts for the respective metals were $Al_2(SO_4)_3 \cdot 16H_2O$, $CdCl_3$, $CaCl_2$, $CrCl_3 \cdot 6H_2O$, $CuCl_2$, $FeCl_3 \cdot 6H_2O$, $HgCl_2$, $NiSO_4 \cdot 6H_2O$, $Pb(NO_3)_2$ and $ZnSO_4 \cdot 7H_2O$. The NTA source was the trisodium salt of nitrilotriacetic acid ($Na_3NTA$). The phosphate source was $H_3PO_4$.

## 2.2 Acclimation Procedures

Batch activated sludge reactors were set up and operated at room temperature on a fill and draw procedure until acclimation. Acclimation was judged to have occurred if 95% of the NTA originally present in complex form had been removed. The procedure used was as follows:

(i) Eight litres of return activated sludge from the Burlington Drury Lane Skyway sewage treatment plant were added to a 20-litre carboy.

(ii) Five litres of primary settled sewage from the Burlington Drury Lane Skyway plant were added and volume adjusted to give a mixed liquor suspended solids (MLSS) level of ≃2 000 mg/L.

(iii) NTA-metal complex was added to give a concentration in the reactor of 16 mg/L as $H_3NTA$.

(iv) Mixture was aerated to maintain a dissolved oxygen level of 3 mg/L, pH was adjusted to 7.5 with NaOH or $H_2SO_4$.

(v) Samples were taken for NTA and other analyses.

(vi) Aeration was continued for 24 hours and samples taken again for NTA analyses.

(vii) If 95% NTA reduction had not occurred, the reactor contents were settled, supernatant decanted and steps (ii) through (vi) repeated.

It was found that the acclimation period for most NTA-metal complexes was less than seven days. However, some complexes required longer acclimation periods, up to 14 days.

## 2.3 Experimental Design

The actual degradation studies were carried out in a single-stage, temperature controlled reactor, which is shown schematically in Figure 1. Acclimated sludge was transferred from the appropriate carboy reactor in sufficient quantity to give a final MLSS of 2 000 mg/L. Primary settled raw sewage was added to give a final volume of eight litres. Metal complexes and supplemental metals were then added and the experiment carried out according to the following procedures:

The experiments were split into two phases, (i) degradation studies on the traditional metals used for phosphorus removal when complexed with NTA (NTA-aluminum-iron and -calcium complexes) and (ii) degradation studies on the remaining heavy metals. In both phases the initial NTA concentrations evaluated were 8, 12 and 16 mg/L as $H_3NTA$. These are representative of levels which could be encountered in wastewater from a community using NTA based laundry detergents. Individual degradation studies were carried out at two different metal levels. The higher metal levels would be representative of a phosphorus removal situation (i.e., $Ca^{2+}$ = 175 mg/L, $Fe^{3+}$ = 15 mg/L and $Al^{3+}$ = 15 mg/L), and the lower levels ($Ca^{2+}$ = 75 mg/L, $Fe^{3+}$ = 4.0 mg/L and $Al^{3+}$ = 2.0 mg/L) more representative of baseline wastewater concentrations. All other metals were studied at 1:1:1 NTA:metal:phosphate ratios. For initial NTA levels of 8 and 16 mg/L, the metal concentrations were as shown in Table 1.

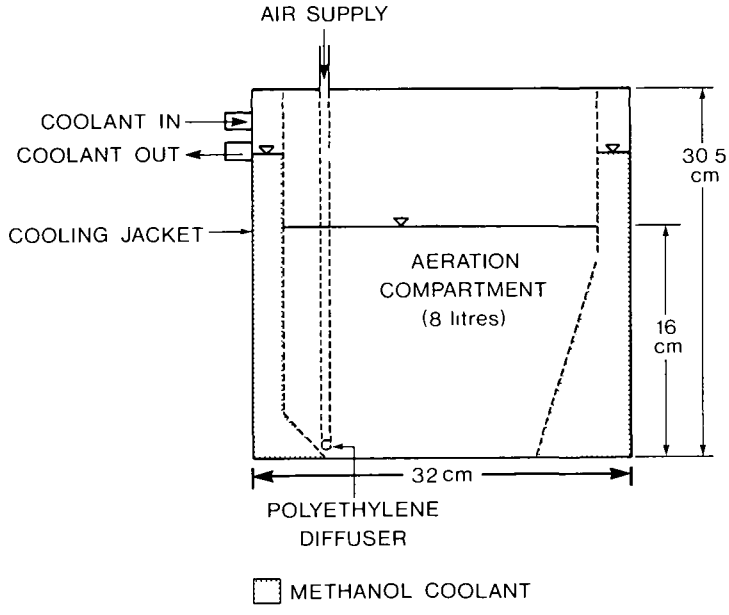


FIGURE 1 CROSS-SECTION OF TYPICAL TEMPERATURE CONTROLLED REACTOR

TABLE 1. METAL CONCENTRATIONS EVALUATED IN DEGRADATION EXPERIMENTS

| Metal | Lower Concentration (mg/L) | Upper Concentration (mg/L) |
|---|---|---|
| $Al^{3+}$ | 2.0 | 15.0 |
| $Cd^{2+}$ | 3.5 | 7.0 |
| $Ca^{2+}$ | 75.0 | 175.0 |
| $Cr^{3+}$ | 1.6 | 3.2 |
| $Cu^{2+}$ | 2.0 | 3.9 |
| $Fe^{3+}$ | 4.0 | 15.0 |
| $Hg^{2+}$ | 6.2 | 12.4 |
| $Ni^{2+}$ | 1.8 | 3.6 |
| $Pb^{2+}$ | 6.4 | 12.8 |
| $Zn^{2+}$ | 2.0 | 4.0 |

## 2.4 Sampling and Analyses

During an experimental run three samples were withdrawn from each reactor at 0, 2, 4, 5, 6, 8, 11, 23, 29, 47, 53 and 71 hours. One sample was filtered and preserved with formaldehyde for NTA analyses. The second sample was used for oxygen uptake measurements, and the third was used for other analyses, including mixed liquor suspended solids. The reactor dissolved oxygen levels were routinely measured two or three times per day. In selected experiments, chemical oxygen demand (COD), total Kjeldahl nitrogen (TKN) and total phosphorus analyses were carried out on the samples. All analyses were carried out according to Standard Methods, APHA et al (1971) or by Standard Technicon Methodology modified for use at the Wastewater Technology Centre. Nitrilotriacetic acid analyses were carried out in accordance with Traversy (1971).

# 3 RESULTS AND DISCUSSION

## 3.1 General

In the evaluation of NTA-metal degradation, 90 batch activated sludge reactor experiments were carried out at initial NTA levels of 8 and 16 mg/L as $H_3NTA$ (a few experiments were run at 12 mg/L). Temperature levels of 5° and 15°C were selected (a few experiments at 10°C). Metal concentrations are shown in Table 1.

The average characteristics of the raw wastewater from the Burlington Drury Lane Skyway plant which was used as feed for the activated sludge reactors are summarized in Table 2.

TABLE 2. AVERAGE CHARACTERISTICS OF BURLINGTON DRURY LANE SKYWAY PLANT RAW WASTEWATER (Jank et al, 1973)

| Parameter | Mean | Range |
|---|---|---|
| $BOD_5$ | 134* | 23 - 280 |
| Filtered TOC | 28 | 16 - 84 |
| Suspended Solids | 187 | 58 - 510 |
| Total Kjeldahl Nitrogen (as N) | 20.5 | 9 - 33 |
| Total Phosphorus (as P) | 5.2 | 1.8 - 11.5 |
| pH | 7.7 | 7.1 - 8.6 |
| NTA (as $H_3NTA$)** | 1.0 | <0.03 - 4.6 |

* All values in mg/L except pH.
**This study.

Oxygen uptake values observed for the reactors during the experimental program ranged from 0.003 to 0.010 mg $O_2$/mg MLSS•hr. The oxygen uptake value for a particular reactor on a given day was largely a function of the initial $BOD_5$ of the raw wastewater. Uptake values in the above range indicated that biodegradation was occurring and there was no significant metabolic interference due to metal toxicity. Only during one experiment with a NTA-mercury complex were oxygen uptake values less than 0.003 mg $O_2$/mg MLSS•hr, indicating metal toxicity to the activated

sludge. Mixed liquor suspended solids during all experiments were maintained at 2 000 ± 250 mg/L.

## 3.2 Degradation of NTA-Metal Complexes

Biodegradation of the various NTA-metal complexes could be approximated by a first-order reaction of the form:

$$NTA_t = NTA_o e^{kt}$$

where: $NTA_t$ = NTA concentration (mg/L) at time t (hrs).
$NTA_o$ = initial NTA concentration (mg/L) at time = 0.
k = NTA degradation rate constant ($hr^{-1}$).
t = time (hr).

Accordingly, the data from the experiments were analyzed and the least-squares fit rate constant (k) determined.

The average degradation rate constants for temperatures of 5°C and 10° to 15°C are presented in Table 3. The number of separate experiments conducted to determine the average degradation rate constants are also indicated. Typical results of a single run at 5°C for the NTA-Ca, -Fe and -Al complexes are presented in Figure 2, for NTA-Pb, -Cr, -Cu and -Zn in Figure 3 and NTA-Cd, -Ni and -Hg in Figure 4. The NTA-metal complexes tested fall into two distinct groups:

(i) readily degradable: NTA-Ca, -Fe, -Al, -Pb, -Cr, -Cu and -Zn; and

(ii) resistant to biodegradation: NTA-Cd, -Ni and -Hg.

These results are in agreement with other studies which have reported limited degradation for NTA-nickel, -cadmium and -mercury complexes.

As expected, the degradation rate constants were strongly influenced by temperature, i.e., considerably lower at 5°C than at 15°C. The few experiments conducted at 10°C indicated little difference from the degradation rates at 15°C. Again these data are consistent with the results of other studies, Eden et al (1972) and Wei et al (1978), in which reduced NTA degradation at wastewater temperatures of less than 10°C was observed.

TABLE 3. FIRST-ORDER DEGRADATION CONSTANTS, $k(hr^{-1})$ FOR SEVERAL NTA-METAL COMPLEXES

| NTA-Complex With | Degradation Constants* | | | |
|---|---|---|---|---|
| | Temp. °C 5 | Number of Experiments | Temp. °C 10-15 | Number of Experiemnts |
| $Ca^{2+}$ | -0.067 | 6 | -0.100 | 3 |
| $Fe^{3+}$ | -0.066 | 6 | -0.102 | 3 |
| $Al^{3+}$ | -0.057 | 6 | -0.075 | 7 |
| $Pb^{2+}$ | -0.072 | 5 | -0.099 | 3 |
| $Cr^{3+}$ | -0.040 | 5 | -0.068 | 3 |
| $Cu^{2+}$ | -0.025 | 4 | -0.072 | 9 |
| $Zn^{2+}$ | -0.051 | 5 | -0.055 | 6 |
| $Cd^{2+}$ | -0.008 | 4 | -0.020 | 1 |
| $Ni^{2+}$ | -0.005 | 5 | -0.021 | 2 |
| $Hg^{2+}$ | 0 | 1 | 0 | 2 |

*Average value.

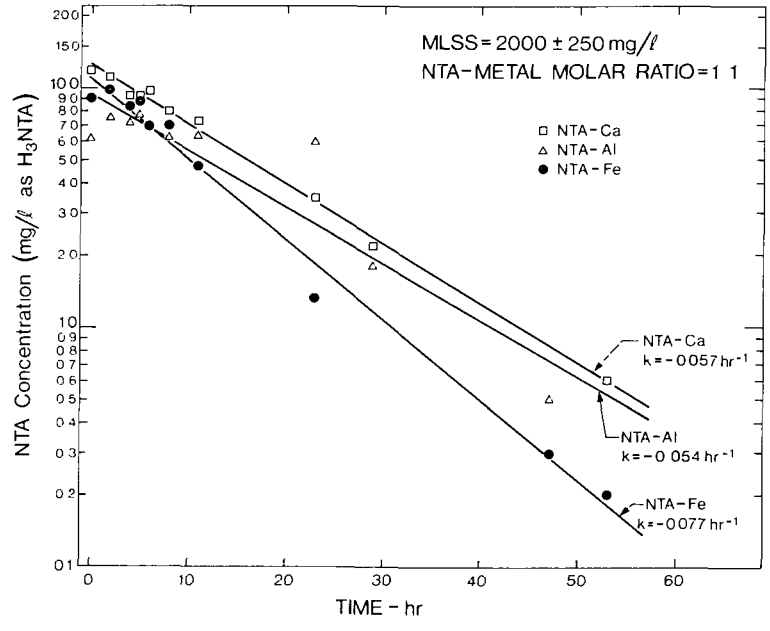


FIGURE 2 TYPICAL DEGRADATION OF NTA-Ca,-Al,-Fe COMPLEXES IN ACTIVATED SLUDGE AT 5°C

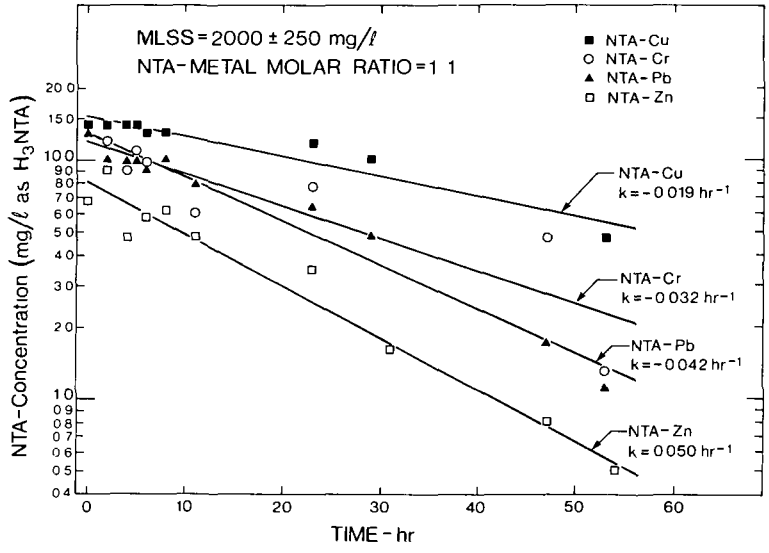


FIGURE 3 TYPICAL DEGRADATION OF NTA-Cu,-Cr,-Pb,-Zn COMPLEXES IN ACTIVATED SLUDGE AT 5°C

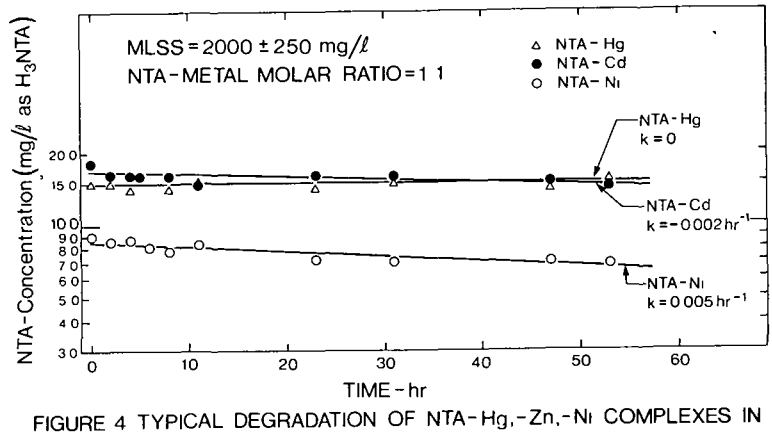


FIGURE 4 TYPICAL DEGRADATION OF NTA-Hg,-Zn,-Ni COMPLEXES IN ACTIVATED SLUDGE AT 5°C

Application of the rate constant data of Table 3 to a conventional activated sludge plant operating under conditions of:

(i) wastewater temperatures of 10° to 15°C;
(ii) initial NTA levels of 10 mg/L as $H_3NTA$; and
(iii) aeration tank residence time of eight hours,

and assuming that all the NTA present is complexed with the particular metal of interest, suggests that overall NTA removal efficiencies for the readily degradable complexes would be in the order of 50%. Removal efficiencies for the bioresistant complexes would approximate 10%. For wastewater temperatures of 5°C, NTA removal efficiencies for the two metal groups would drop to approximately 30% and 5%, respectively. Full scale NTA degradation studies carried out at a conventional activated sludge plant concurrent with these experiments (Wei et al, 1978) demonstrated higher NTA removal efficiencies, i.e., in excess of 80% for wastewater temperatures greater than 10°C and 40% to 50% for temperatures between 8° and 10°C. Although effluent NTA concentrations from treatment plants will definitely be higher during the winter months when wastewater temperatures fall below 10°C, the fact that NTA degradation occurs in receiving streams at water temperatures as low as 2°C (Shannon et al, 1974) and that receiving water temperatures are generally in excess of 10°C for several months of the year, indicates that a buildup of NTA in the aquatic environment is extremely unlikely.

The results of this study show that the NTA-aluminum, -iron and -calcium complexes degrade readily. Consequently, the probably interference of NTA with chemical phosphorus removal systems would be minimal. This has been confirmed in the full scale studies by Wei et al (1978). In the case of calcium, concentrations of calcium present in the wastewater in excess of a 1:1 NTA:metal ratio appeared to increase the degradation rate. This effect is shown in Figure 5. A similar effect for the degradation of NTA-cadmium and -copper complexes has been observed by Walker (1974) for wastewaters having iron levels in excess of 1:1 NTA:iron.

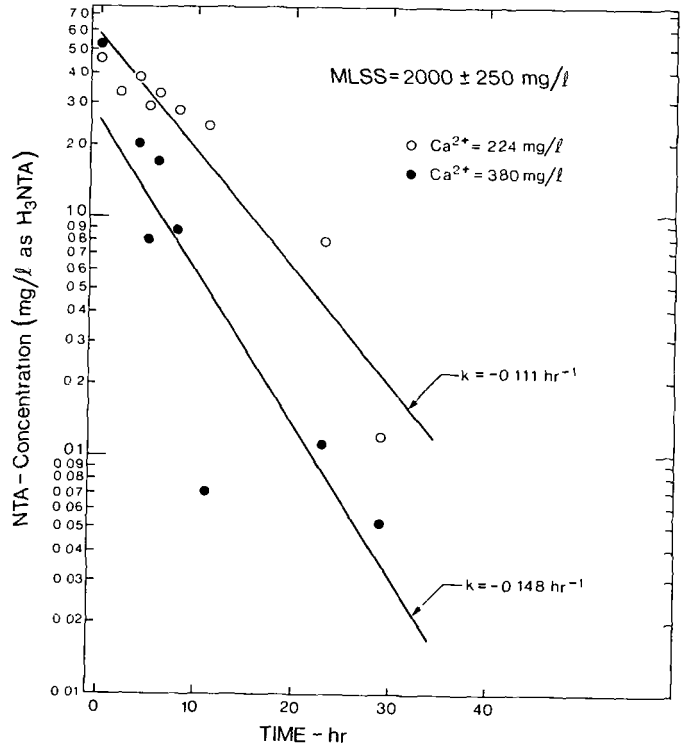


FIGURE 5 COMPARISON BETWEEN NTA-Ca COMPLEX DEGRADATION IN ACTIVATED SLUDGE AT TWO DIFFERENT WASTEWATER CALCIUM CONCENTRATIONS AND 5°C

From this study it would appear that NTA could drastically affect cadmium, nickel and mercury transport through treatment plants, because these complexes were poorly degraded. However, actual conditions which prevail in wastewaters and wastewater treatment plants reduce this possibility because cadmium, mercury and nickel levels in municipal wastewaters are usually very low when compared to other metals such as chromium, zinc and lead. For example, recent analyses by Atkins and Hawley (1978) have shown that even in a heavily industrialized community such as Hamilton, Ontario, the cadmium, nickel, chromium, zinc and lead levels in that wastewater average no more than 0.02, 0.44, 1.2, 8.1 and 1.2 mg/L, respectively. Mercury concentrations are expected to be in the same order of magnitude as cadmium. Thus, the actual levels of Cd and Hg complexes that could exist in wastewaters are very low. Nickel complexes may occur at slightly higher levels, but the majority of the NTA would probably be complexed with other dominant cations such as calcium, iron and aluminum.

This study has shown that NTA-metal complexes with calcium, iron, aluminum, lead, chromium, copper and zinc are readily biodegradable. The poor biodegradability of NTA-metal complexes with cadmium, nickel and mercury appears to be a problem of little practical significance.

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