BIOLOGICAL INHIBITION / TOXICITY CONTROL IN MUNICIPAL ANAEROBIC DIGESTION FACILITIES

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Understanding how to prevent biological inhibition or toxicity in an anaerobic digester is a complex phenomenon that is slowly being understood. Much of the work prior to the 1960's is considered erroneous and misleading because of inadequate experimental techniques and general lack of understanding (1). A major aspect of biological inhibition / toxicity control is in understanding the basic fundamentals of the subject.

BASIC FUNDAMENTALS

First, for any material to be biologically inhibitory or toxic, it must be in solution (soluble form as opposed to non-soluble (particulate) form). If the substance is not in solution, it is not possible for the material to pass through the cell wall and therefore cannot affect the organism.

Second, toxicity is a relative term. There are many soluble organic and inorganic materials which can be either stimulatory or inhibitory or toxic. A good example of this is the effect of ammonia nitrogen on the anaerobic digestion process (see Table 1).

Third is acclimation. When potential inhibitory or toxic materials are slowly increased within the environment, many biological organisms can rearrange their metabolic resources, thus overcoming the metabolic block produced by the normally inhibitory or toxic material. Under shock load conditions sufficient time is not available for this rearrangement to take place.

Finally, there is the possibility of antagonism and synergism. Antagonism is defined as a reduction of the toxic effect of one substance by the presence of another. Synergism is defined as an increase in the toxic effect of one substance by the presence of another. This is an important consideration when designing for potential cation toxicity.

INHIBITORY / TOXIC MATERIALS

Any material, at some concentration level, can be inhibitory or toxic to the anaerobic digestion process. Following is a discussion of the impact of materials in the following seven groups; ammonia, heavy metals, light metal cations, oxygen, short chain organic acids, other organic acids and sulfides.

Ammonia

Source And Entry Into Digester. Whenever a waste biological sludge is thickened above five to six percent prior to anaerobic digestion, ammonia inhibition/toxicity must be considered (2, 3).

Inhibition / Toxic Concentration Levels. Ammonia within the anaerobic digestion process can be in two forms; ammonium ion (NH_4^+) or dissolved ammonia gas (NH_3). Both forms are always in equilibrium with each other, the concentration of each depending on system pH as shown in equation [1].

Equation [1] $$NH_4^+ \leftrightarrow NH_3 + H^+$$

Whenever the system pH is 7.2 standard units or lower, equation [1] equilibrium shifts toward the ammonium ion. At pH values greater than 7.2 standard units, the reaction shifts towards the gas phase. Ammonia gas is inhibitory at a much lower concentration than the ammonium ion. Table 1 lists the effects of ammonia nitrogen concentration on the anaerobic digestion process.

Control. The first step in trying to control potential ammonia toxicity is to analyze the digestion liquid for total ammonia and pH. If the total ammonia concentration is between 1,500 to 3,000 milligrams per liter and the pH is above 7.4 standard units, then there is a possible inhibitory effect due to ammonia gas. This form of ammonia toxicity can be controlled either by dilution of the incoming sludge stream or by the addition of enough hydrochloric acid to maintain the pH between 7.0 to 7.2 standard units. If total ammonia levels are above 3,000 milligrams per liter, then there is toxicity due to the ammonium ion. This form of ammonium toxicity can be controlled by dilution of the incoming sludge stream or depending on how much over 3,000 milligram per liter by the addition of the sodium cation.

Heavy Metals

Table 2 summarizes those substances which are typically considered when discussing heavy metal toxicity in municipal wastewater systems (4). Even though trace amounts of many of the elements listed are necessary for maximum biological development (5), the concentrations which can develop in the wastewater sludge pumped to an anaerobic digester can cause problems. Heavy metal toxicity has frequently been cited as the cause of many anaerobic process failures.

Source And Entry Into Digester. In addition to industrial point sources, heavy metals are also contributed from urban storm water runoff (6, 7), water distribution system corrosion (6, 8) and domestic sewage (8, 9). Table 3 list the results of a U.S. EPA study on the source of the six most commonly found heavy metals in U.S. municipal wastewaters (10). Table 3 also lists the average municipal wastewater treatment plant influent concentrations for the same six metals (11).

Except for nickel and lead, the majority of heavy metals in a municipal wastewater influent are predominately in the particulate form and can be readily precipitated in a wastewater treatment plant. The amount, or percentage, of a particular heavy metal precipitated depends on several factors; pH, concentration of organic material, and the presence of other metals and or pollutants (12-14). In general, sludge will concentrate the metal by a factor of 2,000 to 10,000 times over that of the surrounding liquid, heavy metal concentration (15).

Inhibition / Toxic Concentration Levels. Due to the ease in which heavy metals take part in complex-type reactions with ammonia, carbonates and sulfides, it has been difficult to define a particular total heavy metal concentration which is inhibitory or toxic (16). Table 4 summarizes some information found in the literature. Table 5 summarizes the results from one study showing how chromium, copper and nickel were distributed within an anaerobic digester.

Control. Except for chromium, heavy metal toxicity in anaerobic digesters can be controlled by precipitation with sulfide (18-20). Hexavalent chromium is normally reduced to trivalent chromium which under normal anaerobic digester pH levels is relatively insoluble and not toxic (21).

The reason for using sulfide precipitation is the insolubility of heavy metal sulfides (22). Approximately 0.5 milligram of sulfide is required to precipitate 1.0 milligram of heavy metal. If sufficient sulfide is not available from natural sources, then it must be added in the form of sulfate which is reduced to sulfide under anaerobic conditions.

A potential drawback of using sulfide saturation is sulfide toxicity, production of hydrogen sulfide gas or the generation of weak sulfuric acid which will cause corrosion problems. Because of these potential problems it is recommended that ferrous sulfate be used as the source of sulfide (1). Sulfides would be produced from the biological breakdown of the sulfate, with the excess held out of solution by the iron. However, if heavy metals enter the digester, they will draw the sulfide preferentially from the iron because iron sulfide is the most soluble heavy metal sulfide.

Two other methods of controlling excess sulfide additions have been proposed. One method would be to continuously analyze the digester gas for hydrogen sulfide (19). When there are detectable levels of hydrogen sulfide, sulfate additions would be terminated. When the level became undetectable, additions would start. A second method is the use of a silver-silver sulfide electrode to measure low levels of soluble sulfides (23). The electrode is calibrated in standard solutions of sodium sulfide of known value to yield a parameter, pS. The definition of pS is defined as the negative common logarithm of the divalent sulfide ion concentration. For example, when S^{-2} is 10^{-5}M, pS would be 5.0.

Light Metal Cations

The five common light metal cations (ammonia, calcium, magnesium, potassium and sodium) can have significant impact on the successful operation of an anaerobic digester. Understanding this role has provided insight into how another set of potential inhibitory / toxic conditions can be avoided.

Sources And Entry Into Digester. Generally the local water supply is of such quality (low to moderately hard) that the wastewater sludge produced at the wastewater treatment plant will have low concentrations of these cations. Significant contributions, enough to cause toxicity, can come from several sources.

1. Discharge from industrial sources.

2. Addition of chemicals in the wastewater liquid treatment system, for example the addition of lime for phosphorous removal.

3. Direct addition of alkaline materials to the anaerobic digester for pH control.

Inhibition / Toxic Concentration Levels. Table 6 lists the individual concentration effects of the five light metal cations on the anaerobic digestion process. Table 7 lists the synergistic combinations for the cations. Studies on synergistic effects revealed the following (1).

1. The concentration of the synergistic cation at which the synergistic effect began was less than the concentration required to produce inhibition if the cation was present alone.

2. Mutual synergism between both cations in a cation pair did not always occur. For example with sodium as the toxic cation, ammonium is a synergistic cation but with ammonium as the toxic cation, sodium will be an antagonistic cation.

3. Low concentrations, on the order of 0.005M to 0.05M, could produce the synergistic effect.

Control. Based on current knowledge whenever inhibition is being caused by an excess of a specific light metal cation, the inhibition can be antagonized by the addition of one or more of the cations listed in Table 8. The studies on antagonistic effects revealed the following (1).

1. Low concentrations, on the order of 0.002M to 0.06M, could produce the antagonistic effect.

2. Mutual antagonism between both cations in a cation pair did not always occur. For example with ammonium as the toxic cation, sodium will be an antagonistic cation but with sodium as the toxic cation ammonium is a synergistic cation.

3. Antagonism by multiple cations was superior to that achieved by a single antagonistic cation.

Oxygen

Methanogenic bacteria are probably the most oxygen sensitive organisms known to man. Some engineers and operators have expressed concern over the possibility of oxygen toxicity when using dissolved air flotation thickeners for sludge thickening. In 1971 Fields and Agardy (32) showed “--- that small additions of air (up to 0.01 volumes per volume of digester contents) would not significantly affect anaerobic digester performance.” This value is several magnitudes higher than the amount of air that would be added from a dissolved air flotation thickening system.

Short Chain Organic (Volatile) Acids

Sources And Entry Into Digester. The short chain organic (volatile) acids are the by-product of the non-methanogenic phase of the anaerobic digestion process.

Inhibition / Toxic Concentration Levels. Prior to the 1960’s it was commonly believed that volatile acid concentrations over 2,000 milligrams per liter was toxic to the anaerobic digestion of wastewater sludge. In the early 1960’s McCarty and his co-workers published results from their carefully controlled studies on this subject (2, 27, 28). The results clearly indicated that volatile acids, at least up to 6,000 to 8,000 milligrams per liter, were not toxic to methane bacteria as long as there was adequate buffer capacity to maintain system pH in the range of 6.6 to 7.4 standard units.

Control. A general increasing concentration of volatile acids indicates some type of environmental stress is being placed on the anaerobic digestion process. The addition of the proper alkaline buffering material, as discussed earlier, will allow the process to continue functioning until the reason for the imbalance has been found and corrected.

Other Organic Chemicals

Sources And Entry Into Digester. A wide range of organic chemicals are used in modern day products. Many of these organic chemicals can and do enter the wastewater treatment system. In a wastewater treatment system they enter an anaerobic digester through the sludge stream.

Inhibition / Toxic Concentration Levels. Little data exists on the concentration levels at which organic chemicals would be inhibitory or toxic to the anaerobic sludge digestion process. Table 9 summarizes the results from three studies (29 - 31). In general research has shown the following.

1. Aliphatic, unsubstituted alcohols, except for methanol and propanol, were not toxic at concentrations up to 1,000 milligrams per liter.

2. Aldehydes inhibit the process at lower concentrations than corresponding acids or alcohols.

3. Chemicals with double bonds between carbon atoms caused inhibition at lower concentrations then chemicals with single bonds between carbon atoms.

4. Amino group substitution did not seem to change relative toxicity.

5. Position of functional group seems to play an important part in the inhibition or toxic effect.

6. Increasing hydrocarbon chain length seems to allow increasing concentrations before inhibition is noticed.

7. There seems to be a synergistic effect between volatile acid concentrations in excess of 500 milligrams per liter and organic chemicals.

Control. The best control is through the development of a sewer use ordinance and establishment of a good working relationship with all local industry.

Sulfides

Sources And Entry Into Digester. Sulfides within the anaerobic digester result from three sources: 1) they are present in the influent wastewater stream and enter with the raw sludge; 2) they are biologically produced within the digester by the reduction of sulfates and other sulfur containing inorganic compounds; and/or 3) they are degradation products of sulfur containing organic materials such as proteins.

Inhibition / Toxic Concentration Levels. Soluble sulfide concentrations above 200 milligrams per liter have been shown to be toxic to the anaerobic digestion process (18). The soluble sulfide concentration within an anaerobic digester is a function of the incoming source of sulfur, the pH, the rate of gas production and the amount of heavy metals to act as complexing agents.

Control. Soluble sulfide levels can be controlled either by addition of iron salts (26) and / or elimination of the source(s) of sulfur containing materials.

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TABLE 1
EFFECT OF AMMONIA NITROGEN ON ANAEROBIC DIGESTION (2,3)

Ammonia Nitrogen (NH_3-N) Concentration, milligrams per liter	Effect
50 - 200	Beneficial
200 - 300	No adverse effects
1,500 - 3,000	Inhibitory at pH over 7.4 to 7.6 standard units
Above 3,000	Toxic

TABLE 2
ELEMENTS NORMALLY THOUGHT OF WHEN DISCUSSING HEAVY METAL TOXICITY IN ANAEROBIC DIGESTERS (4)

Consistently Found	Frequently Found	Occasionally Found
Cadmium	Arsenic	Aluminum
Chromium	Iron	Cobalt
Copper	Manganese	Molybdenum
Lead	Mercury	Selenium
Nickel	Silver	Tin
Zinc		

TABLE 3
SOURCES OF SIX COMMON HEAVY METALS FOUND IN INFLUENTS OF MUNICIPAL WASTEWATER TREATMENT PLANTS (10, 11)

Element	Average Percent Contributed, domestic	Average Percent Contributed, non domestic	Average Concentration Of Metal In Wastewater Treatment Plant Influent, milligrams per liter
Cadmium	15	85	0.0055
Chromium	17	83	0.128
Copper	81	19	0.141
Lead	52	48	0.074
Nickel	23	77	0.106
Zinc	55	45	0.430

TABLE 4
SOLUBLE HEAVY METAL CONCENTRATIONS INHIBITORY TO THE ANAEROBIC DIGESTION PROCESS

Heavy Metal	Soluble Concentration, milligrams per liter
Arsenic	0.5 to 1.0
Cadmium	0.01 to 0.02
Chromium (hex)	1.0 to 1.5
Copper	0.5 to 1.0
Nickel	1.0 to 2.0
Zinc	0.5 to 1.0

TABLE 5
HEAVY METAL DISTRIBUTION WITHIN AN ANAEROBIC DIGESTER (17)

Heavy Metal	Heavy Metal Dosage, milligrams per liter	Total Heavy Metal Concentration In Digester, milligrams per liter	Heavy Metal Fractions – milligrams per liter			
			Soluble	Non-soluble	Extra-Cellular	Intra-Cellular
Chromium (+3)	1200 – P	41 – 158	0.065 – 0.11	10 – 97	0.20 – 0.35	25 – 120
	500 – S	53 – 412	0.065 – 0.20	10 – 156	0.09 – 0.14	35 – 224
	100 – S	57 – 106	0.065 – 0.10	18 – 33	0.10 – 0.14	34 – 80
Chromium (+6)	1500 – P	49 – 232	0.065 – 0.25	3 – 55	0.14 – 0.18	48 – 91
	300 – S	49 – 274	0.65 – 0.065	13 – 146	0.09 – 0.14	30 – 126
	100 – S	46 – 84	0.065 – 0.065	1 – 23	0.09 – 0.14	34 – 72
Copper	500 – P	15 – 59	0.036 – 0.036	5 – 34	0.05 – 0.07	6 – 20
	100 – S	11 – 73	0.036 – 0.036	4 – 48	0.05 – 0.12	6 – 18
	20 – S	16 – 22	0.02 – 0.03	7 – 12	0.10 – 0.17	9 – 10
Nickel	250 – P	5 – 31	0.12 – 0.42	2 – 23	0.04 – 0.26	3 – 10
	50 – S	4 – 42	0.14 – 1.22	1 – 31	0.02 – 0.23	2 – 8
	10 – S	4 – 9	0.12 – 0.53	2 – 6	0.05 – 0.11	1 – 3

P = pulse feed to digester
S = step feed to digester

TABLE 6
STIMULATORY AND INHIBITORY CONCENTRATIONS OF LIGHT METAL CATIONS TO THE ANAEROBIC DIGESTION PROCESS (24, 25)

Cation	Stimulatory milligrams, per liter	Moderately Inhibitory, milligrams per liter	Strongly Inhibitory Or Toxic, milligrams per liter
Ammonium	50 - 200	1,000 - 1,500	3,000
Calcium	100 - 200	2,500 - 4,500	8,000
Magnesium	75 - 150	1,000 - 1.500	3,000
Potassium	200 - 400	2,500 - 4,500	12,000
Sodium	100 - 200	3,500 - 5,500	8,000

TABLE 7
SYNERGISTIC LIGHT METAL CATION RELATIONSHIPS IN AN ANAEROBIC ENVIRONMENT (1)

Toxic Cation	Synergistic Cation
Ammonium	Calcium, magnesium, potassium
Calcium	Ammonium, magnesium
Magnesium	Ammonium, calcium
Potassium	None
Sodium	Ammonium, calcium, magnesium

TABLE 8
ANTAGONISTIC LIGHT METAL CATION RELATIONSHIPS IN AN ANAEROBIC ENVIRONMENT (1)

Toxic Cation	Antagonistic Cation
Ammonium	Sodium
Calcium	Sodium, potassium
Magnesium	Sodium, potassium
Potassium	Ammonium, calcium, magnesium, sodium
Sodium	Potassium

TABLE 9
PUBLISHED CONCENTRATION LEVELS OF SELECTED ORGANIC CHEMICALS IMPACTING ANAEROBIC SLUDGE DIGESTION PERFORMANCE

	Concentration Of Organic Chemical In digester – milligrams per liter		
	Unacclimated System		Acclimated System
Organic Chemical	First Notice Reduction In Activity	Fifty Percent Reduction In Activity	First Notice Reduction In Activity
Acetaldehyde	0 – 2 (29)	350 -400 (29)	
Acetic acid	4,000+ (29)		
Acetone	4,000+ (29)		
	1,000+ (30)		
Adipic acid	4,000+ (29)		
Acrolein	5 – 10 (29)	10 – 15 (29)	
	5 – 10 (30)	20 – 50 (29)	
Acrylic acid	5 – 10 (29)	800 – 900 (29)	
Acrylontrile	5 – 10 (29)	150 – 200 (29)	
	<10 (30)	90 – 100 (30)	
Allyl alcohol	<100 (30)	1,000+ (30)	
NOTE: Inhibition level decreases with increasing volatile acid concentration			
1-amino, 2 propanol	125 – 150 (29)		
4-Amminobutynic acid	4,000+ (29)		
Aniline	40 – 50 (29)	2,400 – 2,500 (29)	
Benzoic acid	225 -250 (29)		
1-Butanol	4,000+ (29)		
	1,000+ (30)		
sec-Butanol	4,000+ (29)		
	<100 (30)	1,000+ (30)	
tert-Butanol	4,000+ (29)		
	<100 (30)	1,000+ (30)	
sec-Butylamine	4,000+ (29)		
Butyraldehyde	100 – 200 (29)	2,800 – 3,000 (29)	
Butyric acid	4,000+ (29)		
Catechol	1,000 – 1,200 (29)	2,500 – 2,600 (29)	
3-Chairo-1,2-propandiol	15 – 25 (29)	600 – 660 (29)	
Chloroform	Trace (30)		
1-Chloropropane	30 – 40 (29)	125 – 150 (29)	
1-Chloropropene	Trace (29)	5 – 10 (29)	
2-Chloropropionic acid	200 – 300 (29)	700 – 800 (29)	
Crotonaldehyde	5 – 10 (29)	400 – 450 (29)	
	<5 (30)	50 – 100 (30)	180 – 200 (30)
Crotonic acid	4,000+ (29)		
Dextrpse	1,000+ (30)		
Diethylamine	40 -50 (30)	300 – 500 (30)	
Diethylene glycol	<10 (30)	1,000+ (30)	
N,N-dimethylaniline	1,000+ (30)		
Dodecane	1,000+ (30)		

TABLE 9 (continued)
PUBLISHED CONCENTRATION LEVELS OF SELECTED ORGANIC CHEMICALS IMPACTING ANAEROBIC SLUDGE DIGESTION PERFORMANCE

Organic Chemical	Concentration Of Organic Chemical In digester – milligrams per liter		
	Unacclimated System		Acclimated System
	First Notice Reduction In Activity	Fifty Percent Reduction In Activity	First Notice Reduction In Activity
Ethyl acetate	4,000+ (29)		
	1,000+ (30)		
Ethyl acrylate	300 – 350 (29)	1,200 – 1,300 (29)	
	<10 (30)	300 – 500 (30)	15 -20 (30)
Ethyl benzene	40 – 50 (29)	300 -325 (29)	
	1,000+ (30)		
2-Ethyl-1-hexanol	1,000+ (30)		
Ethylene diamine	15 -20 (30)	100 -200 (30)	
Ethylene dichloride	150 200 (30)		
Ethylene glycol	900+ (30)		
Formaldehyde	5 – 10 (29)	50 – 60 (29)	
	<10 (30)	50 – 100 (30)	
Formic acid	2,000 – 2,300 (29)		
Fumaric acid	1,000 – 1,200 (29)		
Glutaric acid	80 – 100 (29)		
Glycerol	4,000+ (29)		
Hexanoic acid	550 – 600 (29)		
Hydroquinone	400 – 500 (20)		
Isobutyric acid	4,000+ (29)		
Isophorone	1,000+ (30)		
Kerosene	<50 (30)	500+ (30)	
Lauric acid	40 – 50 (20)	200 – 225 (29)	
Maleic acid	200 – 300 (29)		
Methanol	900 – 1,000 (29)	4,000 (29)	
Methyl acetate	4,000 (29)		
Methyl ethyl ketone	4,000+ (29)		
Methyl isobutyl keytone	<10 (30)	100 – 150 (30)	
2-Methyl-5-ethyl pyridine	<50 (30)	90 – 100 (30)	<50 (30)
Nitrobenzene	Trace (29)		
Phenol	40 – 50 (29)	2,400 – 2,500 (29)	
	<50 (30)	350 – 400 (30)	
Phthalic acid	4,000 (29)		
Propanal	500 – 600 (29)		
Propanol	100 – 150 (29)		
2-Propanol	4,000+ (29)		
Propionic acid	700 – 800 (29)		
Propylene glycol	4,000+ (29)		

TABLE 9 (continued)
PUBLISHED CONCENTRATION LEVELS OF SELECTED ORGANIC CHEMICALS IMPACTING ANAEROBIC SLUDGE DIGESTION PERFORMANCE

	Concentration Of Organic Chemical In Digester – milligrams per liter		
	Nonacclimated System		Acclimated System
Organic Chemical	**First Notice Reduction In Activity**	**Fifty Percent Reduction In Activity**	**First Notice Reduction In Activity**
Resorcinol	500 – 600 (29)	3,000 – 3,200 (29)	
Sodium acrylate	<50 (30)	500+ (30)	50 (30)
Sodium benzoate	260 -300 (30)		
Sorbic acid	4,000+ (29)		
Succinic acid	4,000+ (29)		
Tetralin	1,000+ (30)		
Valeric acid	4,000+ (29)		
Vinyl acetate	300 – 350 (30)	900 – 1,000 (30)	

(29) = reference indication