

CHEMICAL CONTROL OF HYDROGEN SULFIDE FROM ANAEROBIC SWINE MANURE. II. IRON COMPOUNDS

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The effectiveness of ionic and metallic iron for control of H_2S evolution from anaerobic swine manure was investigated in laboratory and bench-scale trials. Results indicated that ferrous salts virtually eliminated H_2S evolution. Metallic iron in powdered form was almost as effective, but when added as rods and pipes, was ineffective. Ferric iron was only partially effective. The iron compounds exert their effect on H_2S evolution by precipitating soluble sulfides in the liquid manure as insoluble from sulfides.

INTRODUCTION

A major component of the characteristic odor of liquid swine manure stored anaerobically is a large group of compounds containing sulfur (Barber and McQuitty 1974). Of particular significance is hydrogen sulfide (H_2S), since this gas has not only an offensive odor but is also highly toxic. The possibilities of reducing H_2S evolution from anaerobic swine manure using chemical oxidizing agents have been reported previously (Barber and McQuitty 1975). Another approach to the problem may be to attempt to control H_2S evolution through the use of iron compounds to precipitate out dissolved sulfur compounds in the anaerobic manure.

Gaseous H_2S in the atmosphere surrounding anaerobically fermenting manure arises as a result of the desorption of dissolved sulfides from the manure solution (Barber and McQuitty 1974). The rate at which this desorption occurs and the concentration of gaseous H_2S that may accumulate is determined, according to Henry's Law, by the concentration of dissolved H_2S in the manure solution. In an aqueous medium, dissolved H_2S exists in equilibrium with two other forms of sulfides, namely bisulfide ions (HS^-) and sulfide ions (S^{2-}). The concentration of each form of sulfide is dependent, to a large extent, on the pH of the media. However, this equilibrium may be altered by providing conditions that favor the formation and/or existence of one form over the others (Barber and McQuitty 1974). The approach under question in this paper involves decreasing the equilibrium concentration of dissolved H_2S by precipitating soluble sulfide ions as insoluble metal sulfides. A schematic illustration of the reactions reported above is presented in Fig. 1.

Several researchers have presented evidence for the precipitation of dissolved sulfides by metal compounds. Miller (1950 a,b) found that precipitates of lead, zinc, antimony, bismuth, cobalt, nickel, cadmium and iron could be obtained in culture

media where sulfate reduction was occurring and where those metals were added to the media in ionic form. Miller reported that removal of H_2S by precipitation did not appear to affect the rate of growth of sulfate reducers, but did increase the total amount of sulfide produced. Similar results have been obtained by Wainwright (1971) who showed that certain trace metals greatly increase the total yield of sulfides by sulfate-reducing species of yeast. Pandit (1971) and Pankhurst (1968) have reported that the addition of iron salts to sulfide-containing media effectively prevents the evolution of H_2S gas from the media.

Sulfides also may be precipitated by iron ions released to solution by the corrosion of metallic iron. Starkey (1958) estimated that 2.5% of all steel and iron in service in the United States corrodes away every year, and that corrosion in which sulfate-reducing bacteria are implicated probably accounts for a large percentage of the total. Davis (1967) and Pankhurst (1968) reported that sulfate-reducing bacteria are responsible for corrosion of oil-well casings and well-head equipment.

Another case of anaerobic corrosion was reported during a study involving H_2S production from model stabilization ponds (Gloyne and Espino 1969). In that study, laboratory ponds consisted of iron drums with plastic interior linings. The plastic coating failed on one of the ponds and excessive corrosion occurred. The iron concentration in the affected pond increased to 3.5 mg/liter compared to 0.2-0.3 mg/liter in the other tests, with practically all the sulfides being precipitated as iron sulfide.

Whereas ionic iron has been used successfully to precipitate sulfides from anaerobic digestors receiving domestic sewage sludge (Aulenbach and Heukelekian 1955; Lawrence et al. 1966), its effectiveness in manure solutions apparently has not been demonstrated. From the standpoint of economics and acquisition of the additive, a more practical alternative in the case of manure solutions might involve the use of iron in the form of powder or larger sheets or bars of

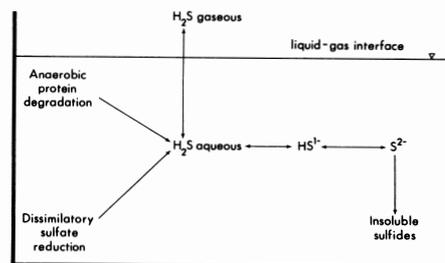


Figure 1. Chemical and biological reactions involving sulfur in anaerobic manure.

metal. However, the rates of corrosion, and consequently the effectiveness with which soluble sulfides are removed from solution, that might be expected upon addition of metallic iron to anaerobic manure apparently are unknown.

The objective of this study, therefore, was to evaluate, by means of a series of laboratory and bench-scale trials, the effectiveness of ferrous salts, ferric salts and various forms of metallic iron for controlling the evolution of H_2S from anaerobic swine manure.

EXPERIMENTAL PROCEDURES AND EQUIPMENT

Laboratory Trials

The investigations reported herein formed part of a larger project involving the evaluation of chemical oxidants, lime, and iron for the control of H_2S evolution from anaerobic swine manure. A total of four trials were conducted, each of approximately 4-wk duration, involving eight different chemical treatments. A description of the iron treatments is given in Table I. The procedure used during the laboratory trials and the characteristics of the manure samples involved have been described previously (Barber and McQuitty 1975).

Each iron treatment was evaluated with manure collected from each of two commercial swine facilities near Edmonton, Alberta. The treated manure samples were incubated

TABLE I DESCRIPTION OF LABORATORY INCUBATION TRIALS

Trial	Manure used	Treatment	Description
I	Installation A (2,000 g)	Control	-
		Ferrous & metallic iron	FeCl ₂ ·4H ₂ O, 1.2 g Powdered iron, 20 g
		Ferric iron Metallic iron	FePO ₄ , 28.4 g Powdered iron, 20 g
II	Installation C (2,000 g)	Control Ferrous & metallic iron	FeCl ₂ ·4H ₂ O, 0.6 g Iron rod, 20 g
		Metallic iron	Powdered iron, 20 g
III	Installation C (2,000 g)	Control Ferric iron Metallic iron	- Fe ₂ (SO ₄) ₃ , 5.3 g Iron rod, 20 g
IV	Installation A (2,000 g)	Control Metallic iron	- Iron rod, 20 g

TABLE II DESCRIPTION OF BENCH-SCALE INCUBATION TRIAL

Digester	Manure sample	Treatment	Description
1	Installation C	Ferrous iron	FeCl ₂ ·4H ₂ O, 158 g
2		Control	-
3		Metallic iron	3-ft section of 2-inch black iron pipe, 2,865 g

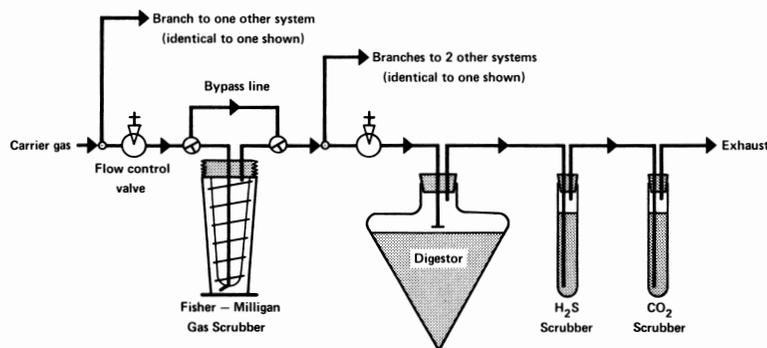


Figure 2. Schematic of equipment used in laboratory incubation trials to monitor gases from fermenting manure.

anaerobically in 2,800-ml laboratory digestors. Nitrogen gas was used to continually flush manure gases out of the digestors and through a two-stage, gas-scrubbing train, thereby enabling the rate of production of these gases to be monitored (Fig. 2). One gas-scrubber contained cadmium acetate solution and the other barium hydroxide solution in which H₂S and carbon dioxide (CO₂), respectively, were precipitated as insoluble compounds. (During this investigation, no distinction was made between various forms of sulfides evolved from manure. For example, carbonyl sulfide and hydrogen sulfide likely both would be trapped in the cadmium acetate absorbent solution. Therefore, throughout this paper, where "H₂S" is used, this should be considered to include other reduced volatile

sulfur compounds as well.) The amount of gas evolved during each collection period was calculated from the weight of dry precipitate collected during that period.

During the first 4 days after the beginning of the incubation period, the amounts of H₂S and CO₂ released from each digester were monitored to ensure that all digestors were behaving similarly. On the 4th day, the chemical treatments were applied. Since the weight of chemical added was not the same in every treatment, sufficient distilled water was added to make a total addition of 100 g to each digester, including the control.

Sixteen to twenty days after the application of the chemical treatments, the contents of each digester were agitated and the amount of H₂S released during the period of agitation was measured. Immediately after

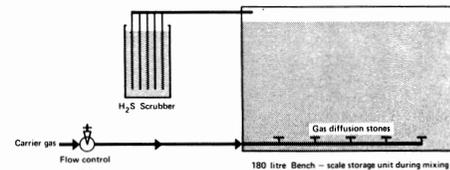


Figure 3. Schematic of equipment used to monitor hydrogen sulfide released from manure incubated in bench-scale digestors.

the cessation of agitation, the contents of each digester were acidified to a pH of approximately 1.0 by the addition of concentrated hydrochloric acid, and purged with nitrogen gas until tests showed that no more H₂S was being released. At pH = 1.0, essentially all sulfides in solution exist as dissolved H₂S and hence are removable by purging the solution with a carrier gas.

Bench-Scale Trial

In a review on anaerobic corrosion of iron pipes in service in gas-field applications, Davis (1967) suggested that one of the requirements for serious corrosion is the coupling of the anaerobic site of sulfate-reducing bacteria development (anode) to cathodic areas that are not completely anaerobic. A trial was conducted, therefore, using three bench-scale digestors, in which the uppermost layers could remain aerobic while the lower layers became anaerobic, to evaluate the effects of metallic and ferrous iron on the amount of H₂S released upon agitation of fermented manure.

The digestors were fabricated from used 250-liter (45-gal) metal drums to which protective plastic coatings were applied. After filling with manure from a nearby swine facility, treatments were applied to each of the three digestors as outlined in Table II. The digestors were allowed to remain undisturbed during the entire incubation period of 30 days. During incubation, the tops were left off the digestors, thereby simulating open manure tanks. At the end of incubation, tops were secured to the digestors and the digestors tipped over as shown in Fig. 3. The contents of each digester were purged for 1 h with nitrogen gas supplied from a pressurized cylinder. The H₂S released was collected in cadmium acetate solution and determined as in the laboratory trials.

RESULTS AND DISCUSSION

The expected effect of adding iron to anaerobically fermenting manure is a reduction in the amount of volatile sulfides released during the fermentation process. Sulfides produced should be bound as insoluble iron sulfide and so should not be released from the manure solution. However, the total sulfide produced from the manure should not be affected adversely, since the bacteria responsible for sulfide production are unaffected by low concen-

TABLE III EVOLUTION OF SULFIDES FROM CHEMICALLY-TREATED MANURE

Trial	Treatment	Sulfides evolved (mg H ₂ S)				
		A	B	C	D	E
I	Control	70.8	132.4	11.6	165.6	380.4
	Ferrous & powdered iron	62.5	12.3	0.0	255.8	330.6
	Ferric iron	84.7	46.3	3.3	20.8	155.1
	Powdered iron	62.5	21.2	0.0	247.2	330.9
II	Control	57.4	89.4	14.4	54.2	215.4
	Ferrous & rod iron	58.3	20.1	5.9	125.5	209.8
	Powdered iron	60.9	44.4	2.6	159.5	267.4
III	Control	39.4	84.5	34.9	62.6	221.4
	Ferric iron	37.0	25.1	7.3	58.0	127.4
	Iron rod	38.9	80.9	21.2	79.4	220.4
IV	Control	68.9	99.1	17.2	143.5	328.7
	Iron rod	69.2	87.1	9.9	156.2	322.4

A = Sulfides released between beginning of incubation and time of chemical additions.
 B = Sulfides released between time of chemical additions and end of incubation.
 C = Sulfides released by agitation at end of incubation.
 D = Total sulfides in digested manure at end of incubation.
 E = Total sulfide production to end of incubation = A+B+C+D.

TABLE IV PERCENT REDUCTIONS IN SULFIDES EVOLVED FROM CHEMICALLY TREATED MANURE COMPARED TO CORRESPONDING CONTROLS

Treatment	Sulfide released after chemical additions†		Sulfide released upon agitation†	
	Manure A	Manure C	Manure A	Manure C
Ferrous & metallic iron	91	78	100	59
Ferric iron	65	70	72	79
Powdered iron	84	50	100	82
Iron rod	12	4	42	39

†Expressed as a percent of the sulfide released during the same period by the corresponding control.

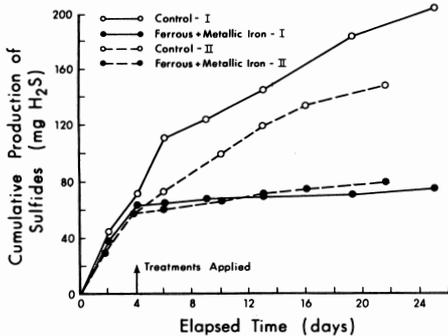


Figure 4a. Hydrogen sulfide production from manure treated with ferrous and metallic iron.

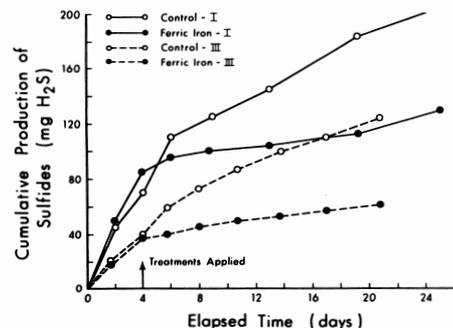


Figure 5a. Hydrogen sulfide production from manure treated with ferric iron.

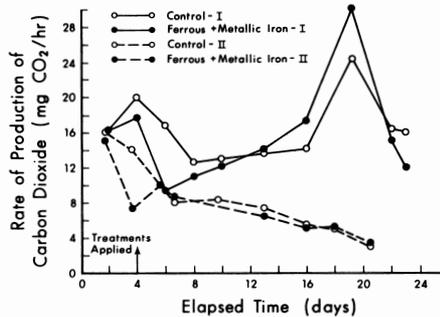


Figure 4b. Carbon dioxide production from manure treated with ferrous and metallic iron.

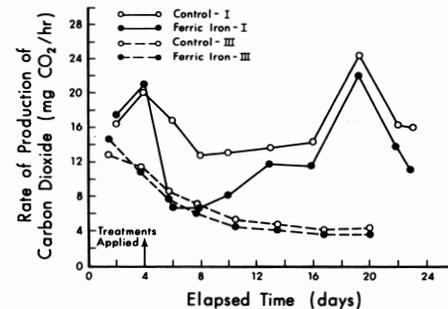


Figure 5b. Carbon dioxide production from manure treated with ferric iron.

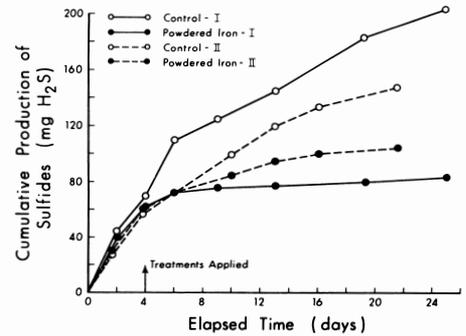


Figure 6a. Hydrogen sulfide production from manure treated with metallic iron added as a powder.

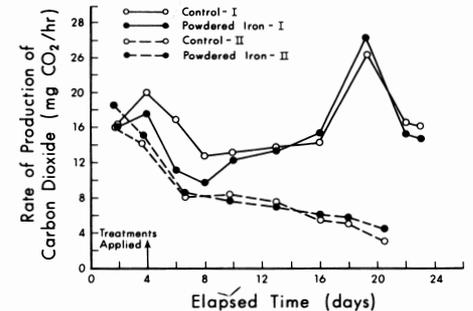


Figure 6b. Carbon dioxide production from manure treated with metallic iron added as a powder.

treatments of iron, both soluble and insoluble, in the media (Aulenbach and Heukelekian 1955; Lawrence et al. 1966). In other words, the concentration of total soluble sulfides in the media should be diminished and the concentration of total insoluble sulfides should be increased relative to an untreated control.

Examination of Figs. 4 - 7, and of the data presented in Table III, indicates that iron added in all four forms reduced the amount of H₂S released from the anaerobically fermented manure during the incubation period subsequent to chemical addition and prior to agitation. This reduction was low and almost non-existent in the case of iron added as an iron rod, but was 50% or more for iron added either in ionic forms or as finely powdered metallic iron (Table IV).

With the exception of iron added in the ferric form, all iron additions resulted in an increase in the total sulfide content of the manure at the end of the incubation period compared to untreated controls (Table III). This suggests that at least some of the sulfides may have been bound as insoluble iron sulfides and so were not released during the incubation period. Further evidence of precipitation of sulfide was provided by the black color of all iron-treated manure.

Except in trials involving ferric iron, the total production of sulfides from iron-treated manure did not differ appreciably from the total sulfide production from the untreated controls (Table III). This observation suggests that addition of iron to the manure had little effect on the bacteria that produce the sulfides. Furthermore, the rates

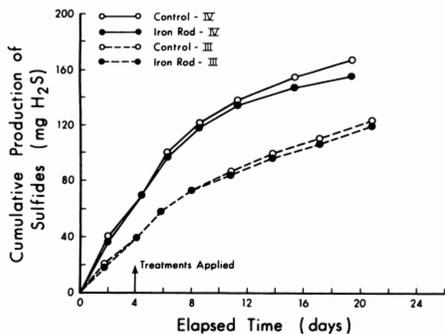


Figure 7a. Hydrogen sulfide production from manure treated with metallic iron added as rods.

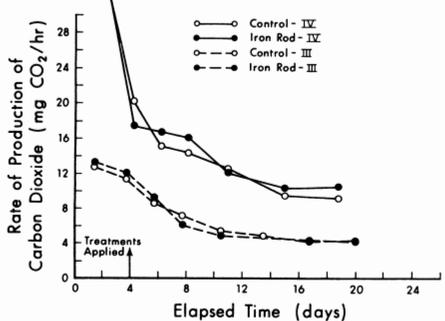


Figure 7b. Carbon dioxide production from manure treated with metallic iron added as rods.

TABLE V RESULTS OF THE BENCH-SCALE TRIAL INVOLVING IRON TREATMENTS

Digester	Treatment	Sulfide released upon agitation (mg H ₂ S)
1	Ferrous iron	40
2	Control	3,640
3	Metallic iron	4,800

of CO₂ production from the iron-treated manure were very similar to those for the corresponding controls (Figs. 4b - 7b), further substantiating the suggestion that iron added either in metallic or ferrous forms does not adversely affect bacterial processes.

Ferric Iron

The addition of iron to manure in the ferric form resulted in lower amounts of total sulfides in the treated manure after digestion and apparently lower total sulfide production as compared to corresponding controls (Table III). Ferric iron is a relatively strong oxidizing agent and, when added to a highly reducing media such as anaerobic manure, will oxidize reduced inorganic compounds while being itself reduced to ferrous iron. In the presence of ferric iron, sulfides in the media most probably would be oxidized to sulfate, hence the lower sulfide content of the treated manure as compared to the control. Because of this

oxidizing effect of ferric ions added to manure, the values presented in Table III for total sulfide production from manure treated with ferric iron are only apparent. The total production in each case was no doubt much higher and quite probably similar to that of the untreated controls.

Examination of Fig. 5b suggests that the addition of ferric iron to manure caused a slight but consistent reduction in CO₂ production, and hence perhaps a decrease in bacterial activity by virtue of a decrease in the pH of the manure.

Metallic Iron

Regardless of the form in which iron is added to a sulfide-containing solution, it probably is converted first to the ferrous form before being precipitated as iron sulfide. In the case of metallic iron, this conversion to the ferrous form involves a slow and difficult process of oxidation which requires some form of catalysis for the reaction to occur at all. In an anaerobic manure solution, the catalytic effect is probably exerted by sulfate-reducing bacteria that serve to depolarize the electrochemical corrosion cells (Starkey 1958). This bacterial corrosion process is no doubt very dependent upon the ease with which the bacteria can come in contact with the zones of corrosion, and hence will occur to a much greater extent if the metal to be corroded has a large surface area. Corrosion of iron rods added to manure during fermentation was evident by deposits of iron sulfide and by pitting of the rods, but because of the relatively small surface of exposure, the rate of corrosion was much less than that of powdered iron, and probably did not provide sufficient ferrous iron for precipitation of the sulfides produced.

Ferrous and Metallic Iron

In the two trials with ferrous plus metallic iron, the objective was to add sufficient ferrous iron to precipitate sulfides already in the media. This was expected to increase the rate of corrosion of the metallic iron. Booth (1964) reported results of laboratory trials in which it was observed that a thin hard protective coating was formed on iron specimens almost immediately upon placement in a medium containing sulfur-reducing bacteria. He observed that the formation of the protective coating could be prevented, and the rate of corrosion increased about sixfold, by the use of a medium containing an excess of ferrous iron to precipitate sulfides as soon as they were produced. In the case of ferrous iron plus iron powder (trial I), enough ferrous iron was added to precipitate 100 mg of sulfide. The difference between the total sulfide contents of the manure in this treatment and in the corresponding control was approximately 90 mg (Table III), suggesting that the entire effect

on sulfide release could have been attributed to the ferrous iron alone.

In the next trial with ferrous and metallic iron (trial II), an iron rod was added instead of powdered iron, with only sufficient ferrous iron being added to precipitate some of the sulfide. The difference between the total sulfide content of the treated manure and the untreated manure was approximately 70 mg in this case (Table III), so that 20 mg of sulfide may have been precipitated by iron released by corrosion of the iron rod. Comparing this 20 mg to the 17- and 13-mg difference between manure treated with iron rods alone and their corresponding controls, a greater rate of corrosion may indeed have occurred in the case of ferrous iron plus the iron rod than with the iron rods alone, in keeping with the expected results as indicated in the previous paragraph.

Bench-Scale Trial

The results of the bench-scale trial with ferrous iron and with an iron pipe are presented in Table V. The only parameter measured was the amount of H₂S released upon agitation of the digested manure. Clearly, the ferrous iron treatment quite effectively reduced the amount of H₂S released upon agitation as compared to the untreated control. However, addition of the iron pipe to the manure had no beneficial effect on H₂S release.

Examination of the pipe revealed that the extent of corrosion was much greater than that which had been observed during the laboratory trials. The part of the pipe which had been in the bottom 6 - 8 inches of the digester was covered with black, flaky deposits, probably iron sulfide, which when scraped off revealed a shiny metal surface beneath. No corrosion had occurred further up from the lower end of the pipe, suggesting that activity of sulfate-reducing bacteria had been limited to that small region at the bottom of the digester. Again, as in the laboratory trials, the rate of corrosion did not appear to be sufficiently great to effectively control H₂S evolution.

Practical Implications

This investigation has shown that the evolution of H₂S from anaerobically fermenting manure is reduced by the addition of ionic or metallic iron to the manure during fermentation. Iron, added as ferrous salts or as a fine powder of metallic iron, effectively prevented the accumulation of soluble sulfides and gaseous H₂S without adversely affecting the bacteria involved in the anaerobic digestion process. Treatment of manure with iron in these forms would appear to be potentially useful in cases where waste liquefaction and organics reduction are desired, such as in anaerobic methane digestion. By precipitating sulfides as insoluble iron sulfides, iron additions to manure in anaerobic digestors could minimize the problems of sulfide toxicity and of

H₂S in the gases collected from the digestors. A toxicity problem could result from repeated applications of ferrous salts if the anions released were toxic to bacteria. This possibility of toxicity is eliminated in the case of iron added in the metallic form and, as such, powdered metallic iron appears to be the best overall alternative for sulfide control during anaerobic digestion. Information concerning the cost of powdering iron for the application was not located by the authors.

The by-products formed as a result of iron treatment are iron sulfides. Sometimes termed "iron pyrites," ferrous sulfides have been used as sulfur fertilizer (Banath 1969) and normally would not be expected to be harmful to soil if treated manure were spread on cropland. In the case of iron being added to manure either as ferrous or ferric chlorides, however, the treated manure could prove harmful to plant growth if it were field-spread, due to the large concentration of chloride salts. Perhaps some difficulty could be experienced with an accumulation of iron sulfides in the bottom of manure storage facilities; however, both laboratory and bench-scale trials indicated that the iron sulfide precipitate remained suspended throughout the manure profile, rather than settling to the bottom. If powdered iron were added on a field-scale basis, some attempt would probably need to be made to add the powder in such a way that an excess of iron was maintained, yet, upon emptying of pits, for example, the unused powder could be reclaimed; however, such a practice seems impractical.

Further Studies

Metallic iron added to manure as a finely ground powder has been shown to effectively prevent the evolution of H₂S from anaerobic manure, whereas metallic iron added as rods or pipes was generally ineffective. However, iron in powdered form is expensive and hence trials should be conducted to determine whether an acceptable balance between the size of metallic iron particles for effective prevention of H₂S evolution and cost may be achieved.

The effectiveness of metallic iron is dependent upon corrosion of the metal. The corrosion process is hastened by the activity of sulfate-reducing bacteria in the manure.

Whereas optimum activity of these bacteria occurs in the pH range of 7-8 (Davis 1967), the pH of iron-treated manure in this investigation was less than 7. Trials could be conducted to evaluate the effect of a combined chemical treatment manure involving liming to pH of 8 and addition of metallic iron.

A technique known as cathodic protection has been employed numerous times in the past to prevent corrosion of buried metal pipes. According to Booth (1964), "In this method of protection the entire structure to be protected is made into the cathode of an electrolytic cell either by connecting to it remotely situated bars of a more electro-negative metal (acting as anodes) or by imposing a direct current between the structure and remotely situated and expendable anodes to which the corrosion is transferred . . ." In the present application, the reverse of cathodic protection might be used to "cause" a large piece of iron or scrap metal to corrode. The technical and economic feasibility of this approach could be investigated in laboratory trials.

SUMMARY AND CONCLUSIONS

The effectiveness of ionic and metallic iron for controlling the evolution of H₂S from anaerobic swine manure was investigated in laboratory and bench-scale trials.

The results of these trials indicate that if large dosages of ferrous salts could be attained economically, H₂S evolution could be vitally eliminated. Powdered iron was almost as effective as ferrous iron; however, metallic iron added to manure as rods or pipes was not found to be effective, although severe corrosion of the metal did occur. Ferric iron was not found to be particularly effective, this apparent ineffectiveness perhaps being due to undesirable pH changes caused by excessive application rates. The iron compounds exert their effect on H₂S evolution by precipitating soluble sulfides in the manure solution as insoluble iron sulfide.

While the economics of the treatment of manure with iron as an odor control agent are not encouraging, such treatment would appear to be potentially useful in cases where waste liquefaction and organics stabilization are required, such as in

anaerobic methane digestion. Further trials are required to study methods for increasing the rate of corrosion of metallic iron in manure solutions.

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