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"BIOX" - A NEW HYDROGEN SULFIDE ABATEMENT TECHNOLOGY FOR THE GEOTHERMAL INDUSTRY

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ABSTRACT

A new liquid phase oxidation process for abatement of hydrogen sulfide emissions from geothermal power plants has recently been developed. The process comprises contacting cooling tower circulating water, in a substantially continuous manner, with a small, substantially less-than-stoichiometric amount of an oxidizing biocide. The oxidizing biocide, in combination with oxygen, converts dissolved hydrogen sulfide to water-soluble sulfates. Biocide-assisted oxidation (BIOX) prevents secondary emissions of hydrogen sulfide from cooling towers that utilize steam condensate for makeup water. The process can also include combining hydrogen sulfide-containing turbine offgases with the treated cooling tower water to control primary emissions. Demonstration testing and commercial operation of the process in cooling systems employing both direct contact and surface condensers has proven successful. Arsenic in cooling tower drift, nitrogen dioxide emissions and the growth of organisms in the cooling water can also be controlled employing this process. Primary organism control is usually achieved by adding larger amounts of a second (usually non-oxidizing) biocide. The process is compatible with scale and corrosion inhibitor-treated cooling water.

INTRODUCTION

Environmental protection is becoming increasingly important in many industries worldwide, including geothermal heat and electric utilization operations. Hydrogen sulfide produced from geothermal fluids is a well-known and major contributor to air pollution. In the past, hydrogen sulfide was often discharged directly into the atmosphere. However, not only is hydrogen sulfide a nuisance emis on, but it is also a very toxic substance. The emission of hydrogen sulfide into the atmosphere is now strictly regulated in many locations where geothermal fields are situated. In the many countries, geothermal electric generating facilities are required by law to abate hydrogen sulfide emissions as part of their permission for operation (Weres, 1984).

Significant progress in controlling hydrogen sulfide emissions has accrued in the past decade at the Geysers geothermal field (Kenny, et al., 1988). Hydrogen sulfide abatement systems developed at the Geysers geothermal field, and elsewhere, generally consist of incineration and liquid redox sulfur recovery processes (Dalrymple, et al., 1989). Gas streams (hereafter referred to as primary emissions) can be incinerated, oxidized over catalysts or absorbed in solutions that are eventually oxidized to yield elemental sulfur and water. Steam condensates (hereafter referred to as secondary emissions) are usually oxidized to also convert hydrogen sulfide to elemental sulfur. More recent technology development at the Geysers has resulted in a combination primary-secondary process that produces thiosulfate as the reaction by-product (Bedell and Hammond, 1987). Various metal catalysts have also been tested for application in geothermal systems (Weres, et al., 1985).

In most brine-dominated geothermal resources, such as those encountered at Salton Sea, abatement of hydrogen sulfide is preferably attacked in the power generation facilities. Attempts to scavenge, scrub or oxidize hydrogen sulfide in the brine have only been partially successful. Oxidation of brine at Salton Sea to convert H_2S to sulfur or higher oxidation state products can result in the formation of ferric iron that is extremely corrosive to piping and deposits as scale with silica (Jost, 1980).

DISCOVERY OF THE BIOX PROCESS

In 1987, Unocal Corp. entered the power generation business when it assumed operation of a small 10 MW power generation facility at the Salton Sea geothermal field. Originally, turbine offgases at the plant were vented to the atmosphere, while steam condensate was utilized for makeup to the cooling tower. Secondary hydrogen sulfide emissions from the cooling tower were controlled fortuitously by zinc-chromate cooling water corrosion inhibitor treatment. Zinc in the corrosion inhibitor precipitated dissolved hydrogen sulfide in the makeup steam condensate as sphalerite, ZnS, that accumulated in the catch basin as sludge.

Upon assuming operation of the power plant and construction of two new power plants since, Unocal changed corrosion inhibitor chemistry to phosphate-based systems due to excessive zinc and chromium sludge formation in the catch basin, and due to an impending ban of the use of chromate in cooling towers by the state of California. The change in inhibitor chemistry resulted in a significant increase in microbiological activity in the circulating water and no more secondary abatement. To mitigate biofouling, organic, non-oxidizing biocides were applied to the water system. Also investigated were the use of chlorine-donor oxidizing biocides to improve microbiocidal efficiencies.

Due to the presence of high concentrations of ammonia in the cooling water (introduced in the hotwell condensate makeup water from the surface condenser, vide post), no attempt was made to achieve breakpoint chlorination. Breakpoint chlorination requires adding an excess of chlorine equivalent to the water to exceed a Cl/N mole ratio > 1.6 to oxidize all of the ammonia to nitrogen gas and to reduce all of the active chlorine-containing species (HOCl, NH₂Cl, etc.) to chloride ion. To achieve breakpoint chlorination in these cooling towers would have required addition of several tons per day of chlorine-containing biocides at costs exceeding several thousand dollars per day.

As the cooling water system was monitored during tests with low dosages of oxidizing biocides, it was discovered that at addition rates of only a few kg per day of chlorine and bromine-stabilized biocide tablets to the cooling tower circulating water in ratios hundreds of times less than stoichiometric amounts required to oxidize hydrogen sulfide, secondary emissions were completely abated by converting dissolved sulfides to soluble sulfate salt (no elemental sulfur solids were observed).

Subsequent jar and pilot cooling tower testing of various oxidizing biocide additions to sulfide-contaminated cooling waters yielded wide ranging results. Oxidizing biocides, such as chlorine gas, sodium and calcium hypochlorites, and chlorine dioxide added to water at up to stoichiometric amounts sufficient to oxidize all sulfides, were only partially successful in converting these sulfides primarily to elemental sulfur. By contrast, compounds such as bromo-chlorohydantoin and trichloroisocyanuric acid added in much less-than-stoichiometric amounts, proved to cause sulfides to rapidly disappear and to produce sulfate ion without precipitating elemental sulfur (Gallup, 1991a).

Based on the success of the process to control secondary emissions by adding much less-than-stoichiometric amounts of the "halogen-stabilized" biocidal agents in tablet form to oxidize all sulfides to sulfate, we investigated the concept of contacting turbine offgas laden with hydrogen sulfide against the oxidizing biocide-treated water to control primary emissions. Bubbling offgas into circulating return water scrubbed the hydrogen sulfide provided the water pH was maintained in the slightly basic range (pH 7 - 8):

$$H_2S_{(g)} = H_2S_{(aq)} \text{ or } H_2S + OH^- = HS^- + H_2O \qquad (1)$$
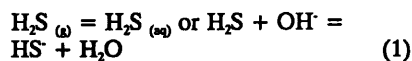
Scrubbed hydrogen sulfide in the offgas was also rapidly oxidized by the biocides to sulfate without formation of elemental sulfur solids (Gallup, 1991b). By-product sulfate in cooling tower blowdown from the BIOX process was re-injected into the geothermal reservoir for disposal.

COMMERCIAL OPERATION OF THE BIOX PROCESS

Surface condensers coupled to gas ejectors are utilized at Salton Sea geothermal field power plants. Steam flashed from Salton Sea hyper-saline brines contain the gases, carbon dioxide, ammonia and hydrogen sulfide. A typical steam analysis is presented in Table 1. Upon condensing the steam, the hotwell condensate consists primarily of ammonium

carbonate/bicarbonate, as shown in Table 2. Non-condensible gases consist primarily of carbon dioxide and hydrogen sulfide (see Table 3). Partitioning of hydrogen sulfide in steam across the condensers is typically 40% to the offgas and 60% to the aqueous phase (condensate) due to solubility effects imparted by ammonia and carbon dioxide (Weres, 1984).

The BIOX process has been utilized since 1989 at Unocal's power generating facilities to control secondary emissions from cooling towers that receive hotwell condensate makeup. The following example of one cooling tower installed at the field is provided for illustration of the process. This example cooling tower is a three-cell, countercurrent Marley unit. Under normal operating conditions, system parameters of the tower are shown in Table 4. Table 5 gives typical cooling tower water analyses. Daily additions of 5 - 10 kg of 7.5 cm trichloroisocyanuric acid tablets are made to the tower forebay for secondary abatement. The tower receives 3.6 kg per hour of H₂S equivalent in the makeup condensate. (In this process scenario, turbine offgas was dispersed over the top of the cooling tower or vented from a stack.) In the absence of an abatement process, the theoretical concentration of H₂S in the cooling tower plumes from secondary emissions should approach 0.7 ppm. Analyses of cooling tower plumes indicate H₂S concentrations typically average < 0.05 ppm, as measured by a Jerome 631 H₂S analyzer (Arizona Instruments, Phoenix, AZ); circulating water analyses show that sulfate ion as measured by a Dionex Model 4500 ion chromatograph (Dionex, Inc., Sunnyvale, CA) is produced at a rate of 11 kg per hour (the theoretical amount of sulfate that can be produced from the sulfides entering the system in the makeup). Thus, secondary abatement as practiced in this tower with the BIOX process approaches 100%.

In 1990, both primary and secondary abatement were achieved in the example tower by compressing offgas from several different turbines and bubbling it into the circulating water at the bottom of the vertical risers to the cooling tower (see Figure 1). The offgas flow rate bubbled into the circulating water averages 1,360 kg per hour, of which 5.5 kg per hour is hydrogen sulfide. Thus the total H₂S equivalent sent to the tower in both the makeup condensate and the offgas is about 9.1 kg per hour. In the absence of abatement, no sulfate ion should be present in the circulating water and the concentration of H₂S in the tower plumes should be about 1.7 ppm. At a dosage of 10 kg per day of trichloroisocyanuric acid tablets added to the catch basin forebay, gas expelled from the circulating return water spray distribution system inside the tower (measured from a standpipe) contained 0.8 kg per hour of H₂S for a primary abatement efficiency of ~85%. In this process option, the tower plumes typically contain 0.15 ppm H₂S and sulfate production in the tower water increases at rates up to 25 kg per hour. Total abatement efficiency is calculated to be ~93%. Therefore, of 218 kg per day of H₂S sent to the cooling tower, only about 19 kg per day is emitted to the atmosphere (Hoyer, et al., 1991).

Recently, commercial testing in the example tower has focused on eliminating the compressor by blowing offgas into a few inches of water in the catch basin and under the packing material in the countercurrent tower. Provided the pH of the circulating water is maintained in the 7 - 8 range (offgas laden with carbon dioxide tends to drive the water pH to the 6 - 7 range) with lime or caustic, excellent primary abatement

continues to be attained. Eliminating the gas compressor from the system and using existing condenser vacuum significantly reduces capital and parasitic load expenses of the process. Scale and corrosion inhibitors, together with non-oxidizing biocides that are compatible with the abatement process, are added to the circulating water. Lime addition for pH control is carefully metered to ensure that the optimum water pH is obtained for efficient scrubbing of the H_2S in the offgas and for mitigating calcium carbonate scaling. Lime dosages in the example presented here are < 50 kg per day.

In 1989 and 1991, pilot and demonstration testing of the BIOX process was successfully conducted at the Bulalo, Philippines geothermal field. This testing was designed to determine if the BIOX process could be applied in a direct contact condenser system utilizing motive steam gas ejectors and a cross-current flow cooling tower. Both primary and secondary abatement were achieved by adding much less-than-stoichiometric amounts of trichloroisocyanuric acid to the cooling tower circulating water (results to be presented at the 14th New Zealand Geothermal Workshop, November 1992).

ABATEMENT CHEMISTRY OF THE BIOX PROCESS

Chlorinated waters or chlorine solutions are used extensively to oxidize hydrogen sulfide in wastewater treatment processes. Hypochlorite generated from applying chlorine to water easily converts sulfides to elemental sulfur:

$$Cl_2 + H_2O = HOCl + H^+ + Cl^- \qquad (2)$$

$$HOCl + H_2S = S + HCl + H_2O \qquad (3)$$
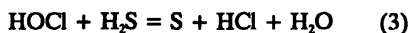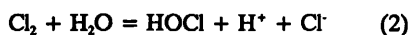
The above chemical reaction has a theoretical requirement of 210 kg of chlorine per 100 kg of hydrogen sulfide. Often, due to the presence of other oxidizable species in wastewater, typical dosage rates range from 500 - 800 kg of chlorine per 100 kg of hydrogen sulfide. Sodium hypochlorite is used to scrub and oxidize hydrogen sulfide to sulfate in many industrial applications. In this reaction, 1750 kg of sodium hypochlorite (bleach) are theoretically required to completely oxidize 100 kg of hydrogen sulfide. Actual bleach requirements can often exceed 2000 kg (Pope and Federici, 1989). Obviously, the use of these oxidants in a typical, geothermal, hydrogen sulfide abatement application would be prohibitively expensive.
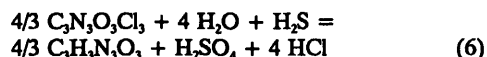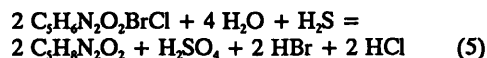
Operation of the BIOX process at the Salton Sea geothermal field in both the primary and secondary abatement modes has shown that dissolved oxygen in circulating water is partially responsible for sulfide oxidation. Typically, only about 1 ppm dissolved oxygen is consumed during the abatement process in each circulation cycle of the cooling tower. Mass balances show that if oxygen were the sole oxidant in the BIOX process, then more than 2 ppm dissolved oxygen would be consumed. Thus, oxygen converts less than 50% of sulfides to sulfate by the following reaction:

$$H_2S + 2 O_2 = H_2SO_4 \qquad (4)$$

The remainder of sulfides in the water are oxidized to sulfate employing much less-than-stoichiometric amounts of oxidizing biocides, implying that these agents react "catalytically", provided that sufficient water is initially present to scrub and "fix" hydrogen sulfide for eventual oxidation.

Due to complexities of the BIOX process in cooling towers, it is difficult to explain why certain oxidizing biocides react in a catalytic fashion at much less than theoretical stoichiometric dosages required to convert sulfides to sulfate. One explanation for the unusual reactivity of bromo-chlorohydantoin and trichloroisocyanuric acid is that these compounds are more stable in water against decomposition than other, more common chlorine-containing oxidants. In this regard, chlorine gas stability in water is improved when added together with cyanuric acid and hydantoin (White, 1972). In the BIOX process developed here, the oxidants are also added as tablets that allow for slow dissolution and "time-release" action.

The oxidizing biocides that have proven most useful in the BIOX process to date are bromo-chlorohydantoin and trichloroisocyanuric acid. The reactions of these oxidizing agents with hydrogen sulfide to yield sulfate are, respectively:

$$2 C_5H_6N_2O_2BrCl + 4 H_2O + H_2S =$$
$$2 C_5H_8N_2O_2 + H_2SO_4 + 2 HBr + 2 HCl \qquad (5)$$

$$4/3 C_3N_3O_3Cl_3 + 4 H_2O + H_2S =$$
$$4/3 C_3H_3N_3O_3 + H_2SO_4 + 4 HCl \qquad (6)$$

To abate 200 kg per day of hydrogen sulfide equivalent sent to a cooling tower at a power plant, even with 50% of the oxidation occurring as a result of reaction with dissolved oxygen, is predicted from Reactions 5 and 6 stoichiometries to require 1420 and 911 kg per day of these oxidizing biocides, respectively. A process to oxidize dissolved sulfides to sulfate in oilfield produced waters at stoichiometric dosages has been developed (Allison and Wimberley, 1987). That < 10 kg per day of these oxidizing biocides is required to effectively convert this amount of hydrogen sulfide to sulfate suggests that the above reactions are somehow catalytic.

Recent kinetic and mechanistic studies have shown that compounds similar in nature to the BIOX agents react rapidly with sulfites to yield sulfates (Yiin, et al., 1987; Yiin and Margerum, 1990). Oxidation reactions of chloramines with sulfites are general-acid-catalyzed over the pH range, 5 - 9, involving H(+) and Cl(+) transfer mechanisms. Further, acid-catalyzed oxidation reactions of organochloramine with organic sulfides to yield organic sulfoxides are well-known (Rees and Sugden, 1973). Thus, it is believed that the oxidizing biocides used in the BIOX process react in a similar, acid-catalyzed manner. The presence of traces of dissolved transition metals in cooling water also appears to "boost" the BIOX reactions. However, regardless of the specific nature of the abatement reaction involved, it has nevertheless been demonstrated that the "BIOX" process developed here is effective in controlling hydrogen sulfide emissions from geothermal power generation facilities.

CONTROL OF NOX AND ARSENIC EMISSIONS

Nitrogen oxides. During development of the BIOX process for hydrogen sulfide abatement at the Salton Sea field, it was discovered that bromo-chlorohydantoin and trichloroisocyanuric acid also assisted in controlling emission of nitrogen oxides from cooling tower waters. Cooling tower

circulating waters at Salton Sea are contaminated with nitrite ion as a result of nitrifying bacteria oxidizing ammonia. The nitrifying bacteria are difficult to control with available biocides. Although much less-than-stoichiometric amounts of the oxidizing biocides were abating hydrogen sulfide emissions, the presence of these agents caused the nitrite ions to be oxidized to nitrate ions.

Salton Sea geothermal brine contains over 500 ppm of ferrous iron. When brine carryover occurs in steam lines, this iron migrates to the hotwell condensate. Ferrous ion then reacts with nitrite when makeup condensate is mixed with circulating water to yield nitrogen oxides:

$$2\ Fe^{2+} + 4\ H^+ + 2\ NO_2^- =$$
$$2\ Fe^{3+} + 2\ H_2O + 2\ NO \qquad (7)$$

$$2\ NO + O_2 = 2\ NO_2 \qquad (8)$$
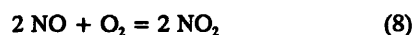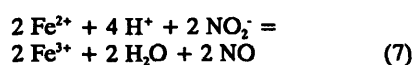
Not only can the nitrogen oxides be emitted from the cooling tower during brine carryover, but also cooling tower blowdown laden with nitrites is often co-mingled with brine in ponds prior to injection into the reservoir (Pye, et al., 1989). By oxidizing nitrite to nitrate with BIOX agents, nitrogen oxide emissions are abated. This is due to the fact that nitrate reacts extremely slowly with ferrous iron to generate nitrogen oxides compared to nitrite. Laboratory studies have shown that nitrite-containing cooling water reacts with iron in brine under a wide range of conditions to generate brown, nitrogen dioxide gas in less than a few minutes. By contrast, when treated cooling water containing primarily nitrate ion is mixed with brine, nitrogen dioxide is not emitted for many days. Reaction kinetics of nitrate and ferrous iron are extremely slow compared to nitrite and ferrous ions (Gallup and Featherstone, 1991).

No emissions of nitrogen oxides to the atmosphere have been detected from Unocal's Salton Sea power plants during commercial operation of the BIOX process (originally developed for hydrogen sulfide abatement). To ensure that nitrogen oxides are abated, nitrite levels in the circulating waters are kept in check not only by adding the oxidizing biocides, but also by adding other non-oxidizing biocides to control nitrifying bacteria and other microbial activity.

Arsenic. Steam produced from many geothermal fields contain traces of arsenic. This arsenic is usually present primarily in the reduced arsenious (+3) state (Ballantyne and Moore, 1988). Where hotwell condensate is employed for makeup to cooling towers, arsenic may be present in circulating waters. For example, arsenic has been detected in Salton Sea, Mak-Ban and Tiwi, Philippines cooling tower waters. Table 6 shows the concentrations of total arsenic and speciation distributions of As^{3+} and As^{5+} in these waters. Determination of the oxidation states of arsenic in the samples was performed by an ion exchange procedure (Criaud and Fouillac, 1989; Ficklin, 1983) and inductively-coupled plasma-mass spectrometry (ICP-MS). Due to oxidation, significant As^{5+} is observed in the cooling waters. In the presence of much less-than-stoichiometric amounts of BIOX agents used for hydrogen sulfide abatement, the ratio of As^{5+} to As^{3+} is dramatically increased. Furthermore, BIOX-treated circulating water contains much less arsenic than untreated water. This is apparently due to precipitation of As^{5+} with calcium,

dissolved iron and/or piping corrosion products to form sparingly soluble calcium and iron arsenates ($CaAsO_4$ and $FeAsO_4$). When the arsenic is precipitated as scale or sludge in the cooling system, the potential for emission of arsenic in cooling tower drift is significantly decreased. At the Salton Sea field, for example, arsenic emissions in drift prior to BIOX commercialization was estimated to be 0.02 kg per day - arsenic was present primarily in the more toxic, +3 oxidation state. Operating the tower with the BIOX process in place reduced the arsenic emissions to 0.004 kg per day, an abatement efficiency of 80%. Further, the arsenic expelled from the cooling tower in the drift was almost exclusively present in the less toxic, +5 oxidation state (Gallup, 1991c).

BENEFITS OF THE BIOX PROCESS

The BIOX process developed here for hydrogen sulfide abatement at geothermal power plants is very cheap to operate due to low addition rates of chemicals. The process has been found to be beneficial in controlling nitrogen oxides and arsenic emissions in addition to abating H_2S. Oxidizing biocides and pH control additives, such as caustic soda and lime, added to circulating water can cost much less than other chemicals used in abatement processes. The BIOX process can also utilize very little equipment. Only when turbine offgas must be compressed to allow sparging in circulating water lines that are under pressure is any capital expense for equipment installation required. In most applications, operating expenses (primarily chemical costs) should be competitive with other, currently-available abatement technologies. BIOX technology is available for license from Picheney World Trade, Inc., Brandeis Division, Greenwich, CT.

BIOX is also an attractive abatement technology with respect to waste generation. No elemental sulfur is produced in the process that would need to be disposed of or sold for relatively low value. Additionally, there are no metal wastes generated by the process. The by-product of the process is soluble sulfate that can be simply disposed in cooling tower blowdown. At the Salton Sea field, blowdown is injected via a dedicated well to assist in re-charge of the reservoir.

ACKNOWLEDGEMENT

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Table 1. Composition of Salton Sea Steam (ppmw)

CO₂	4650
H₂S	50
NH₃	250
N₂	5
H₂	10
CH₄	50
Total NC Gas	5015
As	<0.01
B	2
Hg	<0.001

Table 2. Composition of Steam Condensate (ppmw)

As	0.01
B	3
Hg	0.0005
Fe	0.01
Na	0.1
Cl	1
CO₃	360
NH₃	270
OH	50
HS	35
pH	9.2

Table 3. Composition of Non-condensible Gas

mole %	
CO₂	99.50
H₂S	0.31
NH₃	0.01
N₂	0.01
H₂	0.30
CH₄	0.31
Traces (ppm) of other hydrocarbons	

Figure
"BIOX" PROCESS

Compressor

NC
Gas

From
Condenser

Circulation Water
to Condensers
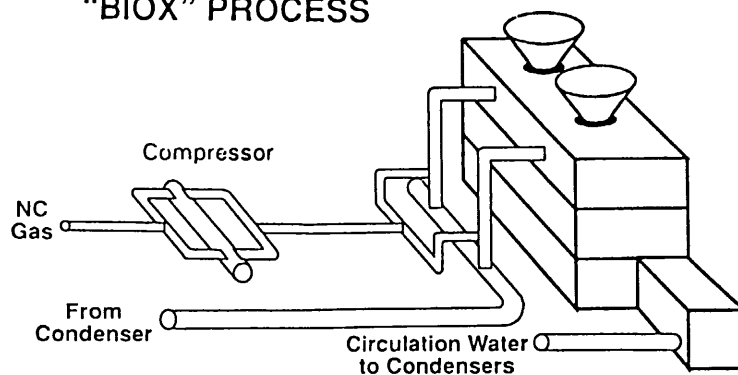
Table 4. Cooling Tower System Parameters

System Volume, L	1,514,000
Air Flow Rate, kg/min	87,130
Recirculation Rate, L/min	119,200
Evaporation Rate, L/min	2,230
Makeup Rate, L/min	2,380
Blowdown Rate, L/min	50
Cycles of Concentration	16

Table 5. Composition of Cooling Water (ppm)

pH	7.2
Ca	80
HCO_3	33
Cl	200
NH_3	800
NO_2	20
NO_3	1,530
SiO_2	3.5
HS	<0.05
SO_4	1,160

Table 6. Arsenic Analyses (ppm)

Location	As(Total)	As^{3+}	As^{5+}
Salton Sea (No BIOX)	0.49	0.40	0.09
Salton Sea (with BIOX)	0.11	0.02	0.09
Mak-Ban	0.50	0.35	0.15
Tiwi	1.7	1.4	0.3