Removal of H₂S using a new Ce(IV) redox mediator by a mediated electrochemical oxidation process

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Abstract

BACKGROUND: Hydrogen sulfide (H₂S) from industrial activities and anaerobic manure decomposition in commercial livestock animal operations is an offensive malodorous and toxic gas even in small concentrations, causing serious discomfort and health and social problems. The objective of this study was to employ for the first time a novel, attractive, low cost, environmentally benign mediated electrochemical oxidation (MEO) process with Ce(IV) as the redox catalyst for H₂S gas removal from an H₂S–air feed mixture.

RESULTS: The influence of liquid flow rate (Qₗ) from 2–4 L min⁻¹, gas flow rate (Q₉) from 30–70 L min⁻¹, H₂S concentration in the H₂S–air feed mixture from 5–15 ppm, and Ce(III) pre-mediator concentration in the electrochemical cell from 0.1–1 mol L⁻¹ on H₂S removal efficiency were investigated. Both liquid and gas flow rates influenced the removal efficiencies, but in opposite directions. Nearly 98% H₂S removal was achieved when the concentration of Ce(IV) mediator ion in the flowing scrubbing liquid reached 0.08 mol L⁻¹.

CONCLUSIONS: The new MEO method proved promising for H₂S removal, achieving high removal efficiency. Integration of the electrochemical cell with the scrubber set-up ensured continuous regeneration of the mediator and its repeated reuse for H₂S removal, avoiding use of additional chemicals. Since the process works at room temperature and atmospheric pressure utilizing conventional transition metal oxide electrodes more commonly used in industrial applications, it is also safe and economical.

INTRODUCTION

The development of processes for the removal of hydrogen sulfide from the atmosphere has been stimulated by the increasing importance of the control of pollution by hydrogen sulfide. H₂S gas is not only malodorous, but also toxic, and even in small amounts it can be fatal to both animals and human beings. It also has detrimental effects on many industrial catalysts: it is a major source of acid rain when oxidized to sulfur oxide on combustion, and it is also highly corrosive both in gaseous form and in solution. H₂S is produced from many industrial activities including the paper industry, petrochemical plants and natural gas refineries. The anaerobic decomposition of sulfur-containing organic and inorganic matter as in wastewater treatment plants is yet another source for H₂S emission. Bacterial growth during storage, agitation and spreading of manure in animal or pig farms releases large quantities of odorous sulfur compounds (H₂S, methyl mercaptan, dimethyl sulfide and dimethyl disulfide) with H₂S showing the highest concentration and emission rate. H₂S contributes substantially to the total odour, since its odour threshold limit is very low ranging from 0.0005–0.3 ppm. Note that the toxicity threshold of this compound is about 10 ppm. Thus, strict environment regulations are continually being strengthened throughout the world to lessen the odour problem, and several H₂S removal methods have been developed for this purpose.

The conventional methods for H₂S removal include fixed-bed solid adsorption, precipitation, liquid biofiltration, alkali aqueous solution absorption, amine aqueous solution absorption, liquid redox processes using soluble oxidants or solid catalysts, a method based on ceria-mixed oxide composites as high temperature desulfurization sorbents, etc., with each method having specific advantages and associated disadvantages. Among the numerous methods developed for H₂S removal, the liquid redox method has been studied extensively in recent times using several soluble oxidants, namely NaOCl, H₂O₂, Cl₂, O₃, etc., in wet scrubbing columns, because liquid scrubbing is economically attractive when the volume of odorous gas to be treated is greater than 5000 m³ h⁻¹ compared with other methods which are suitable for treating the removal of low concentrations of H₂S. Liquid scrubbing of gases involves bringing the odorous gas stream into intimate contact with the scrubbing liquid. A liquid scrubber needs to be well designed to ensure adequate contact between the gas and liquid phases. However, the general
problem encountered in the wet scrubbing systems utilizing various chemicals is that they are consumed in the stoichiometric amount of gases removed, necessitating the use of large amounts of expensive chemicals, which also further add to environmental pollution.

The above problem is circumvented by using electrochemical methods, since a clean reagent ‘electron’ is used in redox processes. Winnick and his coworkers reported an electrochemical process for H₂S gas removal from natural gas. The prototype cell consisted of an electrochemical membrane separator, impregnated with a molten electrolyte, in between the anode and the cathode with a stream of H₂S-containing process gas passing the two electrodes at 650 °C. The electrochemical removal process involved reduction of H₂S at the cathode to H₂ and sulfide ion (Equation (1)), migration of S²⁻ ion through the electrolyte for its oxidation at the anode to elemental sulfur (Equation (2)).

$$
H_2S + e^- \rightarrow H_2 + S^{2-} \quad (1)
$$

$$
S^{2-} \rightarrow 1/2 S_2 + 2 e^- \quad (2)
$$

The reaction kinetics at both the cathode and anode were rapid enough (0.1 A cm⁻²) to be negligible for rate-limiting, and the removal rate of H₂S was found to be limited either by gaseous diffusion from the fuel stream to the cathode–electrolyte interface or by liquid diffusion of sulfur ions through the electrolytic membrane, depending on operating conditions and cell design. However, as the process was operated at high temperatures, materials of stringent specification were required. For example, eutectic mixtures of sulfides or carbonates of alkali metals as electrolyte, porous MgO or lithiated Al₂O₃ ceramic membrane or yttria-stabilized zirconia as separator, porous graphite or porous Ni or lithiated Y₀₀.₉Ca₀₀.₁FeO₃ or CoS₂ as cathode, and porous graphite or porous Ni or lithiated NiO as anode were used.

On the other hand, laboratory scale studies by Kelsall et al. revealed that HS⁻ on electro-oxidation on a gold electrode at pH = 9.3 formed a multilayer of sulfur and soluble oxidation products at 25 °C; however, the elemental sulfur on reduction gave polysulfide ions (Sₙ²⁻; (n = 2–5)), rather than HS⁻. Ateya et al. studied the electrochemical removal of hydrogen sulfide from polluted brines at room temperature using porous carbon felt flow-through electrodes. Formation of elemental sulfur on oxidation was observed, and the reaction rate and the removal efficiency increased with sulfide concentration, potential, temperature and electrolyte flow rate.

In this work, a novel process for H₂S gas removal combining the benefits of liquid scrubbing with the advantages of an electrochemical method has been demonstrated using the mediated electrochemical oxidation (MEO) process with Ce(IV) in 3 mol L⁻¹ HNO₃ as the redox catalyst. The MEO process has been identified as one of the most promising future technologies by the United Nations Environmental Programme (UNEP) for the ambient temperature destruction of toxic pollutants and waste streams. This technology uses the absorption of the pollutant species from the gas phase into a liquid electrolyte, where a metal ion in a high oxidation state is utilized as a mediator to oxidize the dissolved component, and the oxidized form of the mediator is continuously electro-generated in a divided/undivided cell in a closed cycle. Integration of the electrochemical cell with a scrubber column ensures that the reagent used to capture H₂S can be regenerated and reused, thereby avoiding the problem of using fresh oxidant for removal. Although several metal ion based redox systems (Ag(II)/Ag(I), Ce(IV)/Ce(III), Co(III)/Co(II), Mn(IV)/Mn(III)) are available for use as mediators in MEO systems, the Ce(IV)/Ce(III) redox couple was chosen in the present studies for H₂S removal, because this redox system was found to be highly effective in the electrochemical treatment of waste biological materials as well as in the removal of NOₓ and SO₂ from waste gases. Interestingly, the Ce(IV)/Ce(III) redox system was used successfully for the destruction of organic pollutants in aqueous media, and it was found to possess excellent catalytic activity towards organic oxidation. Consequently, the aim of the present work was to study the oxidative removal of H₂S by Ce(IV) oxidant in a scrubber under different experimental conditions to establish the performance of the Ce(IV)/Ce(III) based MEO process for removal of gaseous odour.

**MATERIALS AND METHODS**

**Materials**

Cerium(III) nitrate (Ce(NO₃)₃, 6H₂O, 99.9%) from TERIO Corporation, China, nitric acid (HNO₃, 60%) from Sam Chun Chemicals, Korea, and ferrous sulfate (FeSO₄·7H₂O) from Junsei Chemical Co., Ltd, Japan were used as received without further purification. Nafion® 324 membrane was purchased from Dupont, USA, and mesh-type Pt-coated-Ti and Ti plate electrodes were from Wescos, Korea. All the solutions were made using water purified by reverse osmosis (Human Power III plus, Korea). H₂S cylinder gas (2% H₂S with 9% N₂) supplied by P.S. Chem. Co., Ltd, Korea, and moisture-free air from air compressor supplied by Kyungwon Co., Korea were used.

**Apparatus and process**

The experimental set-up used for H₂S removal consisted of two main components: an electrochemical cell reactor and a wet scrubber column. A schematic diagram is shown in Fig. 1. The electrochemical cell, used for Cel(IV) electro-generation, was a plate-and-frame type narrow gap divided flow cell configuration with a mesh type Pt-coated-Ti anode and Ti cathode separated by a Nafion 324 membrane. The inter-electrode gap was maintained at 5 mm with the help of two Vitron rubber gaskets (thickness = 2 mm). The electrode assembly set-up was tightly clamped to Ti end plates 5 mm thick with the help of a series of Teflon plates (thickness = 5 mm) and rubber gaskets. Two such cells arranged in series were used in the present work. Provisions were made for separate channels by which the anolyte and catholyte solutions flowed across the respective electrodes. A 2.0 L solution of cerium (III) nitrate in 3 mol L⁻¹ nitric acid and a 1.0 L of 2.0 mol L⁻¹ sulfuric acid, stored in separate anolyte and catholyte glass tanks, were continuously circulated through the anode and cathode compartments of the electrochemical cell at 2 L min⁻¹ constant flow rate using a magnetic pump (Pan World Co., Ltd, Taiwan). The electrolysis for generation of the Cel(IV) redox mediators was conducted galvanostatically by applying a constant current of 10 A from a locally made constant current source (Korea Switching Instrument). The effective surface area of each electrode exposed to the solution was 224 cm².

The H₂S removal system was composed of H₂S and an air supply, scrubbing liquid tank, the scrubber column, a data logger and gas analysis systems. The glass scrubber column (ID = 10 cm, height = 160 cm) was filled with 25.4 mm diameter Tri-pak packing. The H₂S–air mixture, obtained by controlled the mixing of air and H₂S using mass flow controllers (MFC), was introduced at the bottom of the scrubber at a constant gas flow rate, and the
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Figure 1. Schematic view of experimental set-up and details of electrochemical cell design used in this study for continuous removal of H$_2$S from H$_2$S–air mixture by Ce(IV)/Ce(III)-based mediated electrochemical oxidation process.

A scrubbing solution (3 mol L$^{-1}$ HNO$_3$ with Ce(IV)) was introduced at the top of the scrubber in a counter-current flow pattern at a constant liquid flow rate. The outlet scrubbing solution containing the reduced form of cerium (Ce(III)) was passed through the electrochemical cell for Ce(IV) generation, and was further re-circulated.

**Analytical methods**

The outlet H$_2$S gas concentration was measured at regular time intervals by a Sequential Thermal Desorber (STD 1000, DANI Instruments, Italy) and GC/FPD (GC-2010, Shimadzu, Japan). The outlet gas sample was collected for 1 min in an aluminum-layered Tedlar sample bag (Top Trading Co., Korea) which was connected through a Teflon valve to a low volume air sampler (Pulse Pump, AeroVironment Inc., USA) with a flow rate of 1.5–2.0 L min$^{-1}$. After sampling, the bags were immediately analyzed.

The operating conditions of the thermal desorption unity and the GC/FPD are given in Table 1. Before each of the successive measurements, pure air from sampling bags was passed through the STD and GC units under the same operating conditions, as used during H$_2$S measurement, to drain-off the adsorbed traces of H$_2$S from the tubing and column.

Before start of the removal experiments, the electrochemical cell was first run until the Ce(III) to Ce(IV) oxidation conversion attained 80%, followed by measuring Ce(IV) in the anolyte by titrating it potentiometrically with Fe(II). Only then was the scrubbing solution allowed into the scrubber.

| **Table 1.** Operating conditions of sequential thermal desorber (STD) and GC/FPD |
|-------------------------------------|-----------------------------|
| **STD**                             | **GC/FPD**                  |
| Sampling time                       | Detector                    |
| 30 mL min$^{-1}$ × 10 min           | FPD                         |
| Cold Trap temperature               | Column                       |
| Low : –10 °C, High : 250 °C         | DB-1                        |
| Hold time                           | (60 m × 0.25 mm ID × 0.25 μm) |
| 2 min                               | Carrier gas                  |
| Outlet split                        | He 25 mL min$^{-1}$         |
| 10 mL min$^{-1}$ (10 : 1 split ratio) | Air flow rate               |
| Flow path temperature               | 110 mL min$^{-1}$          |
| 200 °C                              | H$_2$ flow rate             |
| Oven temperature                    | 90 mL min$^{-1}$           |
| 50 °C (9 min) → 40 °C min$^{-1}$ → 200 °C | Detector temperature       |
| 250 °C                              | 7.66%                       |

The removal efficiencies were calculated from the inlet and outlet H$_2$S concentrations. All the H$_2$S removal measurements were carried out at 20 ± 1 °C.

**RESULTS AND DISCUSSION**

The reproducibility in the H$_2$S measurements with the STD/GC instrument was tested by measuring five times with 10 ppm samples and the coefficient of variation was 7.66%.
Effect of H$_2$S concentration on H$_2$S removal efficiency

The effect of initial feed H$_2$S was studied from 5–15 ppm at a given gas flow rate of 70 L min$^{-1}$ with the scrubbing liquid of 0.5 mol L$^{-1}$ Ce(III) (80% of it as Ce(IV), see Materials and methods) in 3 mol L$^{-1}$ HNO$_3$ at a liquid flow rate of 4 L min$^{-1}$. Figure 2(A) shows the H$_2$S concentration profile and its removal efficiency with respect to treatment time for the different feed concentrations. It was observed for each concentration that the concentration decreased with time and attained a constant value within 30 min, and remained the same thereafter for an extended time as long as 120 min. The steady state concentration was higher for higher feed concentration. In terms of the removal efficiency, one can see that it increased sharply within 30 min before reaching a uniform value. The final removal efficiency was found to decrease with increase in H$_2$S concentration in the range 5–15 ppm, as shown in Fig. 2(B). While 5 ppm H$_2$S showed an 89% destruction efficiency, this was reduced to 74% for 15 ppm initial concentration.

Regarding the MEO mechanism, the following reactions could be envisaged for the efficient removal of H$_2$S by the scrubbing liquid (Ce(IV) mediator catalyst ions in 3 mol L$^{-1}$ HNO$_3$) in the scrubber.

In the electrochemical cell, the electrochemical generation of Ce(IV) from Ce(III) oxidation occurred at the anode:

$$\text{Ce(III)} \rightarrow \text{Ce(IV)} + e^-$$ (3)

The cathodic reaction was the reduction of H$^+$ ions:

$$2\text{H}^+ + 2e^- \rightarrow \text{H}_2$$ (4)

The anodically generated Ce(IV) mediator ions fed into the scrubber could bring about the H$_2$S removal as a combination of several of the following processes.

The absorption of H$_2$S gas into the liquid phase could be represented as an equilibrium at the gas–liquid interface as follows:

$$\text{H}_2\text{S}_{(g)} \rightleftharpoons \text{H}_2\text{S}_{(l)}$$ (5)

H$_2$S is a week dibasic acid with dissociation constants $pK_{a1} = 7.04$ and $pK_{a2} = 11.96$ at 18°C, and in the liquid phase it dissociates according to the following equations:

$$\text{H}_2\text{S} \rightleftharpoons \text{H}^+ + \text{HS}^- \quad pK_{a1} = 7.04 \quad (6)$$

$$\text{HS}^- \rightleftharpoons \text{H}^+ + \text{S}^{2-} \quad pK_{a2} = 11.96 \quad (7)$$

The scrubbing medium being highly acidic, in the present MEO system (3 mol L$^{-1}$ HNO$_3$) the dissolved H$_2$S gas in solution would be present entirely in the molecular form. The corresponding oxidation reaction that could occur in the bulk solution under this solution condition is:

$$\text{H}_2\text{S} + 8\text{Ce(IV)} + 4\text{H}_2\text{O} \rightarrow 8\text{Ce(III)} + 10\text{H}^+ + \text{SO}_4^{2-}$$ (8)

Therefore the above reaction scheme: solubility of H$_2$S into the solution (Equation (5)), followed by its oxidation (Equation (8)) in the liquid phase was responsible for its efficient removal. Although the solubility of H$_2$S in the liquid phase is moderate, since the Henry’s constant for H$_2$S is 856 Pa m$^3$ mol$^{-1}$ (compared with 2630 Pa m$^3$ mol$^{-1}$ for the freely soluble CO$_2$), the fast irreversible oxidation reaction (Equation (8)), would favour the solubility of H$_2$S by moving the equilibrium (Equation (5)), to the right towards the solubilized species.

Effect of scrubbing liquid flow rate on H$_2$S destruction efficiency

Figure 3 shows the changes in H$_2$S removal as a function of experiment time for different flow rates of the scrubbing liquid ca 0.1 mol L$^{-1}$ Ce(III) + Ce(IV) in 3 mol L$^{-1}$ HNO$_3$ solution. These data were collected for liquid flow rates varied from 2–4 L min$^{-1}$ at a constant gas flow rate of 50 L min$^{-1}$, and H$_2$S concentration of 10 ppm.

One can see here that again the H$_2$S removal efficiency increased with time and tended to reach a steady state after 30 min. It was observed that the higher the liquid velocity the better the hydrogen sulfide removal; as shown in Fig. 3 (inset), the 75% removal efficiency at low liquid flow rate 2 L min$^{-1}$ increased to 90% when the flow rate was increased to 4 L min$^{-1}$. Two kinds of benefit could be associated with the improvement at higher flow rates; one factor was the availability of more of the fresh mediator...
Effect of gas flow rate on H$_2$S destruction efficiency

In a series of measurements, removal of 10 ppm H$_2$S by 0.1 mol L$^{-1}$ Ce(III) + Ce(IV) in 3 mol L$^{-1}$ HNO$_3$ solution was studied at a liquid flow rate of 4 L min$^{-1}$, varying the gas flow rate from 30–70 L min$^{-1}$. The results are summarized in Fig. 4. There was uniform behaviour at the different gas flow rates, with the H$_2$S removal efficiency increasing sharply in the initial stage, reaching a steady state at around 30 min, and remaining constant thereafter. However, in this case the final removal efficiency was highest (ca 98%) at the lowest flow rate studied (30 L min$^{-1}$), and decreased with increasing gas flow rate (77% at the highest flow rate 70 L min$^{-1}$), as shown in Fig. 4 (inset). Note that in the above case the supply of oxidant Ce(IV) was maintained constant. The decrease in the H$_2$S removal at high gas flow rate was expected, since at high gas flow rate the Ce(IV) concentration decreases as there is more H$_2$S to oxidize.

It is of significance to note that Turpin et al.$^9$ reported a similar decrease of H$_2$S removal efficiency with superficial gas velocity ($U_G$) in the presence of sodium hypochlorite oxidant; however, this behavior was considered by the authors as unexpected in view of the increased liquid-side volumetric mass transfer coefficient ($k_{Li}$) with increasing $U_G$. Considering (i) the importance of the chemical reaction (H$_2$S abatement was realized by chemical reactions after H$_2$S had transferred from the gas to the liquid phase), and hence the residence time in the column of the gas, (ii) negligible gas-side resistance, and (iii) the graphically observed empirical relation between $k_{Li}U_G$ and the superficial liquid velocity ($U_L$), it was shown that the removal efficiency was proportional to $U_G^{-0.12}$, suggesting that H$_2$S removal efficiency would decrease with gas flow rate. It is tempting to interpret the present results in terms of a similar mechanism, since both studies used fluids (liquid and gas) circulated counter-currently in the column. However, judging from the fact that the compact scrubber column used by Turpin et al. was of a spray type, whereas in the present studies a packed tower was used, complete measurement of the hydrodynamic and mass transfer parameters (pressure drop within the reactor ($\Delta P$), interfacial area ($a$), $k_{Li}a$-side volumetric mass transfer coefficient ($k_{Li}a$)) for the packed bed column is essential as a function of liquid and gas flow rates for a comprehensive understanding of the H$_2$S removal process in the scrubbing system designed for deodorization. Work is in progress in this direction.

Effect of Ce(IV) concentration on H$_2$S destruction efficiency

Some triax experiments were performed to determine the influence of Ce(IV) concentration on H$_2$S removal efficiency in the packed column. For a given liquid flow rate 4 L min$^{-1}$, the redox ion (Ce(III)) concentration in the electrochemical cell was varied from 0–1 mol L$^{-1}$ in 3 mol L$^{-1}$ nitric acid solution. Figure 5 shows the effect of Ce(IV) concentration on H$_2$S removal efficiency. For these measurements, the concentration of H$_2$S was kept to a value of 10 ppm, and gas flow rate at 70 L min$^{-1}$. There was better removal of H$_2$S at higher Ce(IV) ion concentration. For example, the steady state removal efficiency was 77% at 0.08 mol L$^{-1}$ Ce(IV) concentration, which increased to 95% at 0.8 mol L$^{-1}$ Ce(IV) concentration (Fig. 5 inset), apparently indicating that working with a higher Ce(IV) concentration was more advantageous. It should, however, be noted that a removal efficiency as high as 98% was obtained with low Ce(IV) concentration (0.08 mol L$^{-1}$) at low gas flow rate of 30 L min$^{-1}$ (Fig. 4), revealing that low Ce(IV) concentrations and low gas flow rates were beneficial for efficient H$_2$S removal.

It is of significance to notice in Fig. 5 that a neat 3 mol L$^{-1}$ HNO$_3$ solution devoid of any Ce(IV) also exhibited similar removal behaviour. That is, the efficiency initially increased with time, reaching a maximum at 30 min. However, the maximum efficiency for the HNO$_3$ alone was low, i.e. around 30%, which subsequently decreased with time. This small but noticeable H$_2$S removal (30%) by pure HNO$_3$ may be associated with increased gas solubility in the aqueous nitric acid in the presence of the packing material, which could offer increasing surface area and
thus mass transfer to achieve considerable gas absorption into the acid solution. Comparing this behavior of pure nitric acid with the Ce(IV)-containing nitric acid system towards H2S removal (Fig. 5 inset), clearly shows the remarkably efficient and selective performance of the Ce(IV)/Ce(III) based MEO redox system in nitric acid for successfully removing H2S in the environment at room temperature and pressure.

A comparison of the present MEO process with other electrochemical methods developed for H2S removal (e.g. using a membrane separator integrated molten electrolyte, and using flow-through porous electrodes) clearly indicates the simplicity and superior advantages of the MEO method. The severe problems encountered in handling molten electrolytes, and selection of materials for electrodes and cell housing for use at high temperatures in the method of Winnick et al., and selection of specific types of electrodes in the method by Ateya et al. are completely avoided in the MEO process, since it works at room temperature and atmospheric pressure utilizing conventional transition metal oxide electrodes more commonly used in industrial applications.

**CONCLUDING REMARKS**

The following can be concluded from the present work concerning the application of Ce(IV)/Ce(III) based MEO process for the removal of H2S from an air–H2S mixture containing H2S at concentrations 5–15 ppm. (1) The proposed method proved promising for H2S removal, reaching a high removal efficiency. (2) The present study with different liquid and gas velocities indicated that both altered H2S removal efficiencies, but in opposite directions. (3) It was shown that, for a given gas flow rate, H2S removal increased with increasing liquid flow rate, related to the availability of more fresh Ce(IV) mediator catalyst ion, and an increase in the mass transfer parameters. (4) On the other hand, increasing gas flow rate decreased H2S removal efficiency as a consequence of more H2S to oxidize. (5) There was a dramatic increase in efficiency with low Ce(IV) concentration (0.08 mol L⁻¹) in the flowing scrubbing liquid, reaching 98% at 30 L min⁻¹. (6) Unlike other electrochemical methods developed for H2S removal, which work at high temperatures (≈650 °C) or which require special electrodes, the MEO is more economical and safer, since it works at room temperature and atmospheric pressure, utilizing conventional transition metal oxide electrodes more commonly used in industrial applications.

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