Remediation Technologies of Ash and Soil Contaminated by Dioxins and Relating Hazardous Compounds

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In recent years, contamination of toxic organic compounds such as polychlorinated dibenzo-p-dioxins and dibenzofurans (PCDD/Fs), and polychlorinated biphenyls (PCBs) into combustion/incineration ash and soil has become a serious environmental problem. Many efforts have been made to develop efficient remediation technologies which remove, neutralize and/or decompose such compounds in solid materials. The remediation technologies may be classified into the following three types: biological (bioremediation), physical/chemical and thermal remediations. The present paper introduces several remediation technologies for ash and soil and discusses their present states of development.

KEY WORDS: PCDD/Fs; PCBs; remediation process; ash; soil.

1. Introduction

Dioxins are the common term describing polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs). Other important dioxin-related compounds are polychlorinated biphenyls (PCBs). These compounds are classified as the highly toxic polychlorinated aromatic compounds (PCAs) group. PCDD/Fs and PCBs have a number of particular individual members known as congeners. The PCDDs and PCDFs have 75 and 135 congeners, respectively. Only 7 of 75 PCDDs and 10 of 135 PCDFs congeners are regarded as toxic congeners. They have chlorine substitutions in at least 2, 3, 7 and 8 positions. The most toxic congener in PCDD/Fs is 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD). For PCBs, 13 of the 209 congeners are also regarded to have dioxin-like toxicity. They are PCBs which have four or more chlorines with just one or no substitution in the ortho positions. These compounds show a flat configuration with two rings in the same plane and are called “co-planar PCBs”.

The properties of these compounds are semi-volatile, non-polar, chemically stable, lipophilic (lipid soluble) and hydrophobic (poorly water soluble). For PCBs, the properties also include non-flammability, resistance to oxidation and low electrical conductivity. With these properties, PCBs have been used in many applications such as dielectric fluid in power transformers and various lubricating fluids. It can be also pointed out that PCBs are generally contains PCDD/Fs as trace impurities. The structural formula and typical properties of the compounds are shown in Fig. 1 and Table 1.1)

PCDD/Fs existed in the environment are originated from various “primary” and “secondary” sources (see Table 2).2–4) “Primary” source represents PCDD/Fs directly formed within the process. “Secondary” source represents PCDD/Fs remobilized or recycled from the primary sources. The origin of secondary source of PCDD/Fs may be from single or multiple sources. By considering these sources, the following pathways of PCDD/Fs contamination into soil can be pointed out:

(i) leakage from industrial waste

![Fig. 1. Structural formulas of (a) PCDDs, (b) PCDFs and (c) PCBs.](image)

| Properties                  | 2,2',4,5,5' Pentachlorobiphenyl | Decachlorobiphenyl | 2,3,7,8 TCDD |
|-----------------------------|---------------------------------|-------------------|-------------|
| Molecular weight (g/mol)    | 536.4                           | 498.7             | 322         |
| Aqueous solubility (mol/L)  | $4.7 \times 10^5$               | $1.3 \times 10^9$ | $6 \times 10^8$ |
| Vapor pressure at 25°C (Pa) | $1.1 \times 10^7$               | $6.9 \times 10^9$ | $2 \times 10^7$ |
| Melting point (°C)          | 76.5                            | 305.9             | 305         |

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been often used as fertilizer also contains PCDD/Fs.5) Historically, bioremediation using microorganism has been successfully used to remediate soils, sludges, and ground water contaminated by petroleum hydrocarbons, solvents, pesticides and other organic chemicals. For the remediation of PCDD/Fs and PCBs contaminated soil, however, there are many possible candidates of microorganisms, which are still in the experimental stage and needed to further study for the applications to actual conditions. The bioremediation technologies may be classified into two processes, i.e., biostimulation and bioaugmentation. Biostimulation utilizes microorganism, which originally inhabits the contaminated soil. The process stimulates the growth and metabolic activity of microorganism to degrade and transform the contaminant. It is conducted by injection of key nutrients, oxygen, vitamins, enzyme preparations and relating hazardous compounds.

2. Remediation Technologies

To clean up the ash and soil from PCDD/Fs and PCBs contamination, several technologies have been proposed. Some of them utilize separation or extraction operation followed by a specific treatment of the contaminants. The others utilize destruction or decomposition reaction of the contaminants. According to the dominant mechanisms in separation/extraction and destruction/decomposition processes, they are classified into biological, physical/chemical and thermal remediation.

2.1. Biological Remediation (Bioremediation)

Bioremediation utilizes microorganisms, e.g., bacteria, as well as fungi, e.g., white-rot fungi, to decompose the contaminant. The microorganisms or fungi grow in the contaminated area and utilize the contaminant as an energy source and food. Compared to the other remediation technologies, bioremediation has some inherent complexities. The combination control of oxygen, nutrient, soil condition and temperature is a key to enhance the decomposition.

2.1.1. Microbial Remediation

Historically, bioremediation using microorganism has been successfully used to remediate soils, sludges, and ground water contaminated by petroleum hydrocarbons, solvents, pesticides and other organic chemicals. For the remediation of PCDD/Fs and PCBs contaminated soil, however, there are many possible candidates of microorganisms, which are still in the experimental stage and needed to further study for the applications to actual conditions. The bioremediation technologies may be classified into two processes, i.e., biostimulation and bioaugmentation. Biostimulation utilizes microorganism, which originally inhabits the contaminated soil. The process stimulates the growth and metabolic activity of microorganism to degrade and transform the contaminant. It is conducted by injection of key nutrients, oxygen, vitamins, enzyme preparations and other organic chemicals.
and other chemicals into the soil. On the other hand, bioaugmentation is the process that introduces mixture of foreign microorganisms into soil at that site (in situ) or in a bioreactor (ex situ) to initiate and sustain their decomposition activities.

Several bacteria have been intensively investigated for their degradation ability of PCBs such as Pseudomonas, Alcaligenes and Rhodococcus species. These bacteria require oxygen environment (aerobic) to decompose PCBs. Another study for the bioremediation of PCBs contaminated soil has been undertaken by Fava et al. Their study utilized native bacteria which inhabited in the PCBs contaminated waste site. The result showed that the remediation of PCBs contaminated soil was achieved in a bioreactor by the stimulation of native bacteria through addition of oxygen and biphenyls. Inoculation of the other bacteria, such as Pseudomonas and Alcaligenes species, together with native bacteria also enhances the bioremediation of PCBs. Besides that, addition of cyclodextrin, i.e., chemical substances which increase water solubility of organic compounds, can also enhance aerobic biodegradation of PCBs by native bacteria. Both studies give only qualitative results of depletion percentage of gas chromatogram peaks, as evidences for decomposition of PCBs.

An integrated chemical-biological remediation process has been developed to degrade PCBs in soil and sediment. This treatment consists of the chemical pretreatment of soil using reagents (1 vol% H2O2 and 1 mol/m3 FeSO4) followed by inoculation of Pseudomonas and Alcaligenes species. The degradation ratio of PCBs in the soil was 39% after treatment for 500 h.

2.1.2. Fungal Remediation

White-rot fungi are organisms which can degrade lignin. Lignin is a very complex structure polymer which is found in woody plants. This complexity and irregularity of lignin make it resistant to absorption and degradation by intracellular enzymes. Because of low levels of key sources of carbon, nitrogen or sulfur nutrients, white-rot fungi produce enzyme into extra-cellular environment to degrade lignin. That is called lignin peroxidase. This mechanism also gives the fungi the ability to degrade various environmental contaminants.

In laboratory scale, Takada et al. successfully investigated white-rot fungi for remediation of PCDD/Fs. Two species of white-rot fungi, i.e., Phanerochaete sordida YK-624 and Phanerochaete chrysosporium, were compared under stationary low-nitrogen medium to improve their growth. The degradation by Phanerochaet sordida YK-624 ranged from 40 to 76% for PCDDs and from 45 to 70% for PCDFs.

2.2. Physical/chemical Remediation

In the physical/chemical remediation technologies, treatment of contaminated ash and soil is undertaken by separation and/or decomposition through extraction and/or chemical reactions with respect to PCDD/Fs and PCBs. Supercritical fluid extraction and solvent extraction utilize a separation mechanism to remove contaminant. However, others directly decompose PCDD/Fs and PCBs through chemical reactions, such as supercritical water oxidation and base catalyzed decomposition.

2.2.1. Supercritical Fluid Extraction (SFE) and Supercritical Water Oxidation (SWO)

Supercritical fluids show characteristics between liquid and gas phases. Their attractive feature is an ability to extract organic compounds with heavy molecular weights. The schematic phase diagram of supercritical fluid is shown in Fig. 2. Above the critical temperature and in a specific pressure, the fluid becomes a supercritical phase, which can be applied to the extraction of contaminants. By reducing the pressure from critical condition, the contaminant can be precipitated from the fluids. CO2 is often preferred to be utilized in SFE, since it is non-toxic, non-flammable, relatively inexpensive and that its critical temperature is relatively low. Pure CO2 becomes a supercritical phase at 31°C and 7.50 MPa.

PCBs in soil have been tried to remove by using SFE-CO2. Supercritical fluid of CO2 flows through the fixed bed of the soil at 40°C and 10.1 MPa. In this condition, over 90% of PCBs in soil have been removed. In addition, the result showed that the removal rate in dry soil is higher than the wet soil. The effect of water content in soil has been comprehensively investigated by Chen et al. They report that the addition of about 5 mol% methanol as cosolvent enhanced the extraction of PCBs. Another study of SFE-CO2 with acetone as a co-solvent has been carried out to remediate PCBs contaminated soil. Although spiked soil was used, the study gave a promising result in terms of fractional extraction of PCBs, more than 95%.

In the SWO, oxidizers are applied to oxidize the organic compounds in supercritical water. The critical temperature and pressure of water are 374°C and 22.1 MPa, respectively. The oxidizers are usually air, pure oxygen and hydrogen peroxide. In a laboratory-scale test, SWO has successfully decomposed PCDD/Fs from fly ash through oxidation. The temperature, pressure and time applied are 400°C, 30 MPa and 30 min, respectively. The addition of hydrogen peroxide up to 2.0 mass% increases the maximum decom-
position ratio up to 99.7%. They also concluded that the effect of oxidizers becomes stronger in the order: air, pure oxygen and hydrogen peroxide.

2.2.2. Solvent Extraction

The pilot-scale test of solvent extraction to the PCBs contaminated soil in a hazardous waste site has been reported. In the process, liquefied propane is used to dissolve contaminants over ranges of temperature from 49 to 60°C and pressure from 1.32 to 2.84 MPa, respectively. Figure 3 describes the schematic flow diagram of the process. The process consists of three basic operation: extraction, solid-liquid separation and solvent recovery. After sieving and removing oversize materials (>6.4 mm), the soil and liquid propane are then mixed in an extractor at the optimum mass ratio, 1 : 1.5. The extraction equipment has a pressure vessel with a high-speed rotary mixer. At the end of extraction process, solid–liquid separation is performed by static settling. This extraction/separation cycle is repeated by adding the same amount of liquid propane to the extractor. When the last extraction cycle is finished, water of one or two times of the soil mass is added to make residual propane float up in the extractor. Liquid propane containing extracted organic compounds is withdrawn from the pressurized vessel. Solid–water slurry is then filtered by vacuum filtration. Recovery of liquid propane is proceeded by transferring the propane-contaminant mixture to the expansion tank. In the tank, propane is vaporized in a gas–liquid system under a reduced pressure. The remained fluid, which contains contaminants and water, is drained from the tank. The purified propane is reused after re-liquidification in a compressor. The result of the test showed that removal efficiency of PCBs in soil reached about 91.4 to 99.4%. These values were obtained when applying three extraction/separation cycles.

2.2.3. Base Catalyzed Decomposition (BCD)

BCD remediation is also the technology to decompose and degrade PCDD/Fs and/or PCBs in soils. The decomposition and degradation reactions are promoted by the presence of hydrogen donors at 300 to 350°C. NaHCO₃ is used and decomposed into CO₂, H₂O and Na₂CO₃ in the process. During heating, hydrogen donors generated from organic compounds in soil play a role in dechlorination reaction.

Figure 4 illustrates a schematic flow chart of the process. After sieving and weighing, soil was mixed with 3% NaHCO₃, for PCDD/Fs remediation. The mixture is heated up to 350 to 400°C in nitrogen atmosphere. After heating for about one hour, the fractional removal of PCDDs was more than 99% and PCDDs were not detected in the treated soil. For remediation of PCBs contaminated soil, a pilot-scale test has been conducted. The optimum reaction temperature and concentration of NaHCO₃ were 330°C and 3%, respectively. The fractional removal of PCBs was about 99.9% under this condition.

2.3. Thermal Remediation

Thermal remediation technologies utilize heat to enhance vaporization of the contaminants and their decomposition. Vaporization of PCBs is used in thermal blanket and microwave remediation technologies. PCDD/Fs and PCBs decompose in the incineration and ash-melting processes at relatively high temperatures. Other technologies use heat to promote decomposition reactions, such as thermal dechlorination process and microwave process with an alumina bomb.

2.3.1. Rotary Kiln Incinerator

A type of thermal remediation technology is the so-called Hybrid Thermal Treatment System (HTTS). Figure 5 illustrates the flow diagram of the pilot-scale facilities of the HTTS, which is applied to hazardous waste sites. This is based on the direct combustion mechanism using natural gas as fuel. A portable module with capacity of 10 to 15 Mg/h consists of incinerator, air pollution control system, such as wet, dry or dry & wet system, and other site-specific auxiliary system. The incinerator is divided into two sections, i.e., a rotary kiln furnace and secondary combustion chamber for off-gases from the rotary kiln.

The soil is charged in the rotary kiln furnace. Operation temperature of the kiln is about 620°C. The treated-soil is then quenched, while the flue gas is transported to the secondary combustion chamber after cool down and separation from the ash. In the secondary chamber, flue gas is heated at a temperature higher than 800°C and for a residence time more than 2 s. Water quench operation and alkali scrubber are applied to avoid further PCDD/Fs formation through de novo synthesis. Concerning the air pollution control, wet system is the most effective to reduce PCDD/Fs.
The removal efficiency of trace organic compounds such as PCDD/Fs and PCBs is enhanced with turbulent gas flow applied in the secondary combustion chamber. For PCBs contaminated soil, the decomposition ratio reaches more than 99.99% with the emission of PCDD/Fs lower than the required standard criteria.

2.3.2. Thermal-blanket System

Another thermal remediation for surface of soil contaminated by PCBs is known as the thermal-blanket system. The pilot-test flow diagram is shown in Fig. 6. The technique utilizes heating element assembly covered by a ceramic insulation and an impermeable silicone fiberglass blanket.

Contaminated soil is covered by a thermal-blanket. Operation temperature is set for heating the soil surface ranging from 815 to 925°C. The rate of decomposition of PCBs is dependent on the temperature and soil depth. In the test, target of the soil-depth for removal of PCBs was 15 cm. Temperature of soil surface reached over 800°C with temperature gradient around 32°C/cm. After heating for 24 h, fractional removal of PCBs reached more than 98%. The downward migration of PCBs through soil was not detected. Vaporization plays an important role in the removal of PCBs during the process. PCBs traveled upward in the soil were removed at the surface by applying air flow. Off-gas passes through a thermal oxidizer to decompose PCBs and other hydrocarbon compounds. This flameless oxidizer operates at temperatures between 875 and 925°C with residence time about 0.5 s. In addition, granular activated carbon is used as an adsorbent to back-up the functions of the oxidizer.

2.3.3. Application of Microwave Energy

The application of microwave energy to soil remediation has been attempted. In a pilot-scale facility (see Fig. 7), cylindrical container with the capacity of 1 Mg soil was used to remove trichloroethylene (TCE) as a model contaminant. Principally the microwave energy (100 MHz to 300 GHz) emitted from a generator penetrates the soil and vaporizes water and contaminants. In the container, microwave is generated from an antenna inside the perforated...
polytetrafluorethylene (PTFE) tube placed vertically at the center of the soil layer. Vapors formed from the soil were sucked through the perforated PTFE tube by a vacuum pump. More than 99% of TCE were removed from the soil by 75 h irradiation.

In a laboratory-scale experiment (see Fig. 8), Abramovitch et al. also have attempted to decompose PCBs in soil by using microwave energy. Cu$_2$O or aluminum powder and 10 kmol/m$^3$ NaOH as reductive dechlorination reagents are mixed with soil sample. The mixture is then placed in a sealed quartz container tube, protected by machineable ceramic alumina, which is called alumina bomb. Then alumina bomb is placed inside a household type microwave oven. The reaction in the alumina bomb is promoted by heat generated by microwave energy. The operation temperature can reach 1200 to 1300°C. The decomposition ratios of PCBs were 97.3% for the case using aluminum powder/10 kmol/m$^3$ NaOH and 95.3% for the case using Cu$_2$O/10 kmol/m$^3$ NaOH.

2.3.4. Melting Treatment

The melting treatment with high temperature is generally applied to decomposition of PCDD/Fs in fly ash from municipal waste incinerators. There are several types of furnaces to treat fly ash, such as plasma-melting furnace, rotating type melting furnace and swirling-flow furnace. Each furnace has characteristic that makes very competitive with each other.

In the plasma-melting furnace, ash is melted by plasma-torch of a Cu-electrode non-transfer type. Furnace temperature is more than 1500°C and the decomposition ratio of PCDD/Fs exceeds 99%. This furnace can treat ash at a capacity of around 2 Mg/h under a standard condition. Figure 9 shows the flow diagram of a plasma melting process.

The rotating type surface melting furnace has a double cylindrical structure (see Fig. 10). The burning and melting of fly ash occur in the upper space of furnace, which is called the primary combustion chamber. The outer cylinder and furnace bed rotate at a rate of one revolution per hour and its shape is determined by the angle of repose of the feed material. The ash melts from the surface of the bed and then falls to the secondary combustion chamber. The temperature in the secondary chamber furnace reaches 1300°C and the decomposition ratio of PCDD/Fs is more than 99%. This furnace fueled by a mixture of air and kerosene. Its process capacity is 600 to 670 kg/h.

Figure 11 describes the process flow diagram of the swirling flow furnace and relating facilities which can treat fly ash at a capacity of 1.2 Mg/h. The mixture of butane and air is used as fuel. During operation, temperature in the furnace achieves 1300°C. The result showed that about 98.4% of PCDD/Fs are decomposed.

All of these melting furnaces are equipped with an air pollution control system to avoid further formation of PCDD/Fs and other gaseous pollutants in the post-combustion zone. The plasma melting furnace employs calcium hydrate injection and catalytic denitrification for the flue gas treatment. Gas cooler, scrubber and electrostatic precipitator are used in the surface rotating furnace. For the swirling furnace, the flue gas is treated in secondary combustion.
Fig. 9. Plasma ash melting flow system (simplified figure).

Fig. 10. (a) Flow diagram of the total process, (b) Rotating type surface melting furnace.

Fig. 11. Flow diagram of swirling-flow furnace and relating facilities.
chamber before transported to the cooling chamber and dust separator.

2.3.5. Thermal Dechlorination

A full-scale facility to remove PCDD/Fs in fly ash has been developed using thermal dechlorination reaction at a low temperature. Figure 12 shows the schematic diagram of the process flow. Important process parameters are temperature and retention time of fly ash. They should be 350 to 400°C and 1 to 3 h, respectively. In order to prevent further formation of PCDD/Fs discharging temperature should be set below 60°C. The main equipment which has a capacity of 500 kg/h consists of a reactor and a cooler. Fly ash from municipal waste incinerator is transported into the reactor and heated up by electric heaters. It is then cooled down in a water cooler and discharged to a cement solidification process. A low oxygen condition is maintained by supplying N₂ gas. With the heating temperature at 350°C and residence time in 1 h, the decomposition ratio of PCDD/Fs reached 99.7%.

2.3.6. Thermal Dechlorination

Figure 12. Flow diagram of the low temperature thermal dechlorination process.40)

Table 3. Remediation technologies of ash and soil contaminated by PCDD/Fs, PCBs and related compounds.

| Process | Contaminants | Contaminated media | Operation conditions | Decomposition ratio (%) | States | Ref. |
|---------|--------------|--------------------|----------------------|-------------------------|--------|------|
| Bioremediation : 1. Native bacteria in the PCBs contaminated waste site 2. Integrated chemical-biological 3. White-rot fungi | PCBs | Soil | 28-30 | O₃ | n.a.² | L | [16,17] |
|          | PCBs(2-CB)  PCDD/Fs | Soil | 30 | Air | 39 | L | [18] |
|          | PCBs(2-CB)  PCDD/Fs | In-vitro | 30 | Air | 40-70 | L | [21] |
| Physical/Chemical : 1. SFE CO₂ 2. SFE (CO₂+Acetone) 3. SWO 4. Solvent extraction (lithified propane) 5. BCD | PCBs | Soil | 40 (10.1) | CO₂ | 90° | L | [24] |
|          | PCBs | Soil | 60-80 (21.8-37.9) | CO₂ | > 95° | L | [26] |
|          | PCDD/Fs | Fly ash | 400 (30) | O₂ | 99.7 | L | [27] |
|          | PCDD/Fs | Soil | 49-60 (1.32-2.84) | Propane | 91.4-99.4° | P | [29] |
|          | a. PCBs | Soil | 300-350 | Air | 99.9 | P | [30] |
|          | b. PCDD/Fs | Soil | 350-400 | N₂ | 99 | L | [31] |
| Thermal : 1. Rotary kiln (natural gas) 2. Thermal blanket (electric) 3. Microwave (electric) 4. Plasma (electric) 5. Surface melting furnace rotating type (ceresone) 6. Vitrification by stirring flow furnace (bunace) 7. Low temperature thermal-dechlorination (electric) | PCDD/Fs | Soil | 1400 | Comb. gas<sup>†</sup> | 99.99 | F | [33] |
|          | PCBs | Soil | 800 | Air | 98 | P | [34] |
|          | a. TCE (model) | Soil | 100 | Air | > 99 | P | [35] |
|          | b. PCBs | Soil | 1200-1300 | Air | 95.3-97.3 | L | [36] |
|          | PCDD/Fs | Fly ash | >1500 | Comb. gas<sup>†</sup> | > 99 | F | [37] |
|          | PCDD/Fs | Fly ash | 1320-1340 | Comb. gas<sup>†</sup> | 99.99 | F | [38] |
|          | PCDD/Fs | Fly ash | 1300 | Comb. gas<sup>†</sup> | 98.4 | F | [39] |
|          | PCDD/Fs | Fly ash | 350 | N₂ | 99.7 | F | [40] |

Table 4. Limitation and advantages of remediation technologies.

| Technologies | Limitations | Advantages |
|--------------|-------------|------------|
| Bioremediation | - limited contaminant to degrade - many factors to be controlled | - relatively higher cost - reduced treatment - difficulty to monitor degradation efficiency | - lower degradation rate - difficult to monitor degradation efficiency - less cost under favorable conditions |
| Physical/Chemical | - post treatment of waste leachate or residual reagent - limited treatment of treatment of volatile heavy metals | - produce relatively small amount of harmful waste - more than 99% reduction of contaminant | - larger capacity in-volume of good - higher removal efficiency of the bio solidification process | - available in portable in-site process |

3. Summary

Tables 3 and 4 summarize the development states and
limitations/advantages of the remediation technologies. Most of the thermal remediation technologies seem to be in a more practical stage as compared to the others. There are many options in the thermal remediation technologies to be selected. Physical/chemical remediation technologies may need further efforts to scale up, since there are still many limitations to be broken through such as capacity, corrosion problem and especially relatively higher cost. The development of bioremediation appears to be still in a progressing stage to find out the most suitable bacteria strains and appropriate values of other process parameters. Although, bioremediation seems to produce no harmful waste, required time for the remediation is longer than the other technologies. Table 4 also shows some requirements to develop and/or select remediation technologies. These requirements will be substantial criteria to develop and/or select remediation technologies.

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