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Gallium arsenide (GaAs) leaching behavior and surface chemistry changes in response to pH and O_2

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ABSTRACT

Gallium arsenide (GaAs) is a material widely used in electronic devices. Disposal of electronic waste containing GaAs in municipal solid waste landfills raises concerns about the public health and ecological risks associated with the potential release of toxic arsenic (As) species. In this study, different tests were performed to investigate the leaching behavior of particulate GaAs in aqueous solutions. In the U.S. Toxicity Characteristic Leaching Procedure (TCLP) and California Waste Extraction Test (WET), the concentrations of As released from the GaAs particles were about 2.6–2.8-fold higher than the regulatory limit (5 mg/L). A much higher As concentration (72 mg/L), accounting for as much as 15.4% of the initial As in GaAs, was solubilized in a pH-7.6 synthetic landfill leachate under ambient atmosphere after 120 days. Additional tests performed to evaluate the dissolution of GaAs under a range of redox conditions, pH levels, ionic strength, and presence of organic constituents commonly found in landfills revealed that oxic environments and mildly alkaline conditions (pH 8.1–8.5) promote release of As (chiefly arsenite) and gallium species to the surrounding aqueous environment. The rate of As release in long-term exposure experiments was initially constant but later progressively diminished, likely due to the formation of a passivating layer on the surface of GaAs consisting of corrosion products rich in poorly soluble gallium oxides (Ga_2O_3 and $Ga(OH)_3$). This hypothesis was confirmed by surface analysis of GaAs particles subjected to leaching using X-ray photoelectron spectroscopy (XPS). These findings suggest that further research is needed to assess the potential release of toxic As from electronic waste in municipal landfills.
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1. Introduction

Gallium arsenide (GaAs) is a semiconductor material widely used in electronic devices. It is a crystalline intermetallic solid composed of arsenic (As) and gallium (Ga) with oxidation states ranging between $Ga^{(0)}As^{(0)}$ to $Ga^{(+III)}As^{(-III)}$ (Carter et al., 2003). The superior electronic properties of GaAs make it suitable for the manufacture of light-emitting diodes (LED), as well as microwave and integrated circuits which are widely used in electronic devices such as satellite communication devices, radar systems, solar panels, smart phones (4G technology), and tablets (Golio and Newgard, 2001, Yoon et al., 2010). GaAs is also being considered as a material for the manufacturing of thin film photovoltaic solar modules and other photovoltaic devices (USGS 2014).

The industrial application of GaAs is growing rapidly. The worldwide production of low-grade primary Ga, which increased by an average of 20% per year from 2004 through 2014, was estimated at 440 metric tons in 2014 (USGS, 2017). GaAs is the most important Ga compound in use. In the US, imports of GaAs wafers ($225 million) and Ga metal ($4 million) accounted for all domestic consumption of Ga in 2016 (USGS, 2017). In the same year, integrated circuits and optoelectronic devices were responsible for 60 and 40% of the US consumption of Ga, respectively. The worldwide consumption of GaAs is expected to continue increasing due to the growing wireless telecommunications infrastructure in Asia and the increasing demand for fourth generation (4G) smartphones, which employ up to 10 times the amount of GaAs as second generation (2G) cellular handsets (USGS, 2014). The As and Ga concentration in GaAs field effect transistors (GaAs-FETs) used in 2G mobile phones was reported to average 0.54% and 0.83%, respectively (Uryu et al., 2003).

The increasing utilization of GaAs in electronic devices has raised concerns regarding the fate of this material in the environment, and its potential risks to public and environmental health. In particular, there is potential for release of highly toxic As when GaAs-containing devices are discarded in municipal mixed solid waste (MSW) landfills. According to current regulations, most

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electronic waste (E-waste) is not classified as hazardous waste because of its residential origin. The fraction of E-waste that is disposed of into municipal MSW landfills is high and varies significantly with geographic location (Kiddee et al., 2013; Ongondo et al., 2011). In Australia, 84% (by weight) of E-waste generated in 2008 was landfilled (EPHC, 2009). In the United States, it is estimated that 60% of the E-waste collected was disposed of in MSW landfills or exported to developing countries (US-EPA, 2013).

Reports on the leaching potential of GaAs in MSW landfills are lacking, but elevated concentrations of metals and metalloids (Pb, Ni, As, Fe, Al) that exceeded Australian drinking water guidelines were detected in landfills receiving E-waste, and/or in groundwater impacted by landfill leachates (Kiddee et al., 2014). An study conducted in Japan investigated the leaching of As and Ga from GaAs FETs used in 2G mobile phones using synthetic solutions containing 1 g of sample to 10 g of leaching solution, *i.e.,* nitric acid or ammonium to adjust the pH at values ranging from ca. 2 to 11 (Uryu et al., 2003). The authors reported that the maximum As and Ga concentrations (21 and 15 mg/L, respectively) were detected at pH 2, the lowest pH value tested. Leaching tests have also revealed that the regulatory limit established for Pb in the U.S. Toxicity Characterization Leaching Procedure (TCLP) and/or the limits set for Pb and Ni in the California Waste Extraction Test (WET) were exceeded in leachates from some components of discarded mobile phones (e.g., plastic casing, printed wire boards) and computers (e.g., video cards, random-access memory (RAM), central processing units (CPU)) (Komilis et al., 2013; Li et al., 2009; Yadav et al., 2014, Zhao et al., 2014). The TCLP and the WET tests are standardized methods utilized by the U.S. Environmental Protection Agency (US-EPA) (EPA, 1992), and the California Environmental Protection Agency (Cal/EPA) (CCR, 1991a,b), respectively, to simulate landfill leaching and determine whether a waste material should be classified as hazardous based on its toxic characteristics.

In spite of the increasing utilization of GaAs in electronic devices, the fate of GaAs under landfill conditions has not yet been investigated carefully. Landfill leachates contain a broad range of organic and inorganic pollutants (Kjeldsen et al., 2002; Moody and Townsend 2017) that could potentially enhance GaAs corrosion, leading to the release of highly toxic arsenic. This metalloid is a known carcinogen that has a stringent maximum contaminant level (MCL) of 10 μg As L^{-1} in drinking water. Gallium arsenide has also been classified as an immunotoxicant and a group I carcinogen to humans (Flora and Dwivedi, 2012). Although GaAs is often assumed to be poorly soluble, several toxicology reports have shown that this material can undergo slow oxidation in aqueous solutions containing complexing agents, leading to the release of high levels of As and Ga (Carter et al., 2003; Pierson et al., 1989).

The leaching potential of GaAs is likely to vary depending on the biogeochemical conditions prevailing in municipal MSW landfills. Landfills are characterized by complex biogeochemical conditions as indicated by the sharp changes observed in redox conditions, microbial activity, and leachate pH and composition with landfill age. Upon landfilling, MSW undergoes a complex series of chemical and biological reactions leading to the generation of landfill leachate. Depending on the phase of decomposition, the redox conditions in a landfill can range widely from oxic (aerobic phase) to strict anaerobic (methanogenic phase) (Kjeldsen et al., 2002). Likewise, the pH and composition of the leachate can vary significantly with landfill age, environmental conditions, waste composition and landfilling technology. During the initial acid phase, the leachate is often characterized by low pH values (4.5–7.5, average 6.1), and by high concentrations of easily degradable organic compounds such as volatile fatty acids (VFAs) and other organic acids. In the later stable methanogenic phase, the pH tends to increase to 7.5–9.0 (average pH = 8) and the relative concentration of poorly degradable compounds and ammonium increases dramatically, due to the removal of biodegradable organics (Kjeldsen et al., 2002). The ionic strength of landfill leachate is generally high but it can vary widely depending on the landfill and its age (Kjeldsen et al., 2002; Kulikowska and Klimiuk, 2008).

The objective of this study was to evaluate the stability and leaching behavior of GaAs under simulated landfill conditions. The dissolution of As and Ga was evaluated under a range of redox conditions, pH and ion strength levels, presence and absence of organic constituents commonly found in landfill leachates, as well as in two standardized leaching tests including the U.S. Federal TCLP and the California WET procedures. X-ray photoelectron spectroscopy (XPS) surface analysis and thermodynamic stability studies were undertaken to aid in the interpretation of the observed GaAs corrosion and dissolution patterns.

2. Materials and methods

2.1. Chemicals

GaAs (99.999% purity) was purchased from Sigma-Aldrich (St. Louis, MO, USA). Arsenite (as $NaAsO_2$, ≥ 90% purity) and arsenate (as $Na_2HAsO_4{\cdot}7H_2O$, ≥ 99% purity) were obtained from Fisher Scientific (Waltham, MA, USA), and Ga^{III} (as Ga_2O_3, 99.99%) from Alfa Aesar (Ward Hill, MA, USA).

2.2. Batch leaching experiments

All experiments were conducted in triplicate using glass serum flasks (160 mL) supplied with leaching solution (100 mL) and GaAs (90 mg, particle size 1–2 mm). Flasks were then sealed with butyl rubber stoppers and aluminum crimp seals and their headspace was flushed for 4 min with different gas mixtures according to the purpose of the assay. The impact of different pH levels ranging from 6.8 to 8.5 on the dissolution of GaAs was evaluated in flasks supplied with a mineral medium containing 1 g L^{-1} sodium bicarbonate (medium M1) and air atmosphere (21% O_2). Desired pH levels were set by modifying the CO_2 concentration in the headspace to values ranging from 0 to 16% depending on the experiment. The same mineral medium was utilized in experiments performed to assess the impact of the O_2 content in the headspace (0–87.8%) on GaAs dissolution, both under circumneutral pH (6.7–7.2) and at mildly alkaline pH (8.1–8.5) conditions. Different amounts of pure He, O_2 and/or CO_2 gas were provided to reach the desired O_2 and pH levels. To attain pH 6.7–7.2, the headspace of the flasks was supplied with 20% CO_2 gas, whereas the flasks maintained at pH 8.1–8.5 were not amended with CO_2. In anoxic experiments, the liquid medium was flushed with N_2 for 3 min. Next, the flasks were sealed using butyl rubber septa and an aluminum seals, and the headspace was flushed with a suitable O_2-free gas mixture for 4 min. Assays supplied with resazurin, a redox-sensitive dye, confirmed that this procedure was adequate to provide and maintain anoxic conditions under the conditions of our experiments.

GaAs dissolution was also investigated in a pH-7.6 simulated landfill leachate (medium M2) containing organic and inorganic contaminants (in mg L^{-1}): $NaHCO_3$ (2600), $MgCl_2{\cdot}6\ H_2O$ (946), NaCl (2020), KCl (298), Na_2HPO_4 (142), Na_2SO_4 (71), $CaCl_2{\cdot}2H_2O$ (368), sodium acetate (574), sodium citrate dihydrate (97). This medium was sparged with air for 2 min prior to use in the assays. The headspace of the bottles was flushed with air.

All flasks were incubated in the dark at 30 °C in an orbital shaker (100 rpm). Liquid samples were withdrawn periodically to monitor their pH value, soluble As and Ga concentration, and As speciation. Samples of the headspace were also collected to quan-

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