



Investigation on stability and leaching characteristics of mixtures of biogenic arsenosulphides and iron sulphides formed under reduced conditions

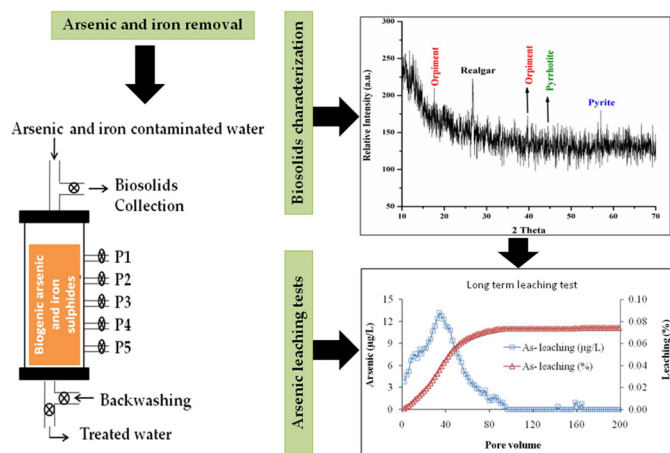
Arvind Kumar Shakya^a, Parasmani Rajput^b, Pranab Kumar Ghosh^{a,*}

^a Department of Civil Engineering, Indian Institute of technology Guwahati, 781039, India

^b Atomic & Molecular Physics Division, Bhabha Atomic Research Centre, Trombay, Mumbai, 400085, India



GRAPHICAL ABSTRACT



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ABSTRACT

Arsenic is removed from aqueous phase through precipitation as arsenosulphides and/or co-precipitation and adsorption on iron sulphides. Studies were carried out to ascertain the stability of reduced biogenic arsenic and iron sulphide precipitates formed in an attached growth reactor (AGR) through a series of experiments based on Toxicity Characteristic Leaching Procedure (TCLP), aging and long term leaching tests. About half of the AGR was initially added with waste activated carbon (WAC) to support the growth of mixed microbial consortia and used for treatment of arsenic and iron contaminated simulated groundwater. The X-ray diffraction (XRD), X-ray absorption near-edge structure (XANES) and extended X-ray absorption fine structure (EXAFS) spectroscopy results indicated that the biosolids were mainly composed of arsenosulphides and iron sulphides. While TCLP and aging tests were conducted in anoxic as well as oxic conditions with the aim to evaluate stability of biomass containing biogenic sulphides, long term leaching test was conducted through supply of aerated distilled water

* Corresponding author.

E-mail address: pkgghosh@iitg.ernet.in (P.K. Ghosh).

to evaluate the stability of spent WAC as well. Results generated from the research indicate that the concentration of leached arsenic never exceeded 123 µg/L under all conditions tested, thus biosolids not imposing an environmental hazard.

1. Introduction

Arsenic removal from drinking water can be achieved through mainly phase transfer, i.e. by changing soluble arsenic into solid phase arsenic. Commonly practiced arsenic removal methods such as adsorption, precipitation, ion-exchange, coagulation-flocculation and electro-coagulation generate oxidised arsenic bearing waste [1]. The oxidised wastes are often associated with re-release of arsenic in reduced environments due to reductive dissolution of iron and other metal based oxides [2–4] and anion exchangers [5].

Biological processes are found to be successful for arsenic removal either as precipitation of arsenic sulphides, co-precipitation and/or adsorption with iron sulphides form acidic waters [6,7], drinking water [8] and landfills [9] in sulphidogenic bioreactors added with iron. The reduced arsenic bearing waste thus generated typically contains arsenic, iron and sulphur in reduced form, which are different from oxidised arsenic bearing waste. Although, reduced arsenic waste is considered to be stable in reducing environment of landfills [10], literature data on the stability of such wastes is limited. Jong and Parry [10] studied the stability of arsenic immobilized by microbial sulphate reduction in a bioreactor treating sulphate and mildly acidic water containing mixture of metals (iron, copper, zinc, nickel, arsenic, magnesium and aluminium). Although arsenic is often found associated with iron, co-occurrence of arsenic along with Cu, Zn, Ni, Al, Fe and Mg in groundwater sources is seldom reported. Arsenic leaching is not so easy in presence of other metal sulphides which may provide adsorption or concomitant precipitation with the other metal sulphides [11]. Thus nature of biosolids generated in a sulphidogenic bioreactor treating arsenic contaminated groundwater will be different from biosolids containing sulphides of many metals. Hence, the study by Jong and Parry [10] does not represent true stability of mixtures of arsenosulphides and iron sulphides. Moreover, arsenic and other metals concentration in acidic waters is reported to be very high [6,11] which is not a condition in drinking water sources. Therefore, further studies are required to evaluate the stability of biogenic metal sulphides generated in sulphidogenic bioreactors treating arsenic and iron contaminated drinking water.

Although, several researchers [2–4], followed different testing procedures such as toxicity characteristic leaching procedure (TCLP), California Waste Extraction Test (CalWET) and aging on arsenic bearing solids, all were oxidized arsenic bearing wastes. TCLP is the most widely used regulatory protocol for testing arsenic bearing wastes [1], it underestimates arsenic leaching from the sludge as it performed under oxidising conditions [12] and shows a large variation in arsenic release values for drinking water wastes [1]. Furthermore, TCLP is a rapid test (18 h duration) that also involves high speed agitation which may not indicate long term arsenic leaching in field applications where there is no physical disturbance over a month or year [10]. Deep subsurface geochemical environment are characterized by metabolic activities of various taxonomic groups which causes redox transformations of arsenic and play major role in arsenic mobility [13–15]. Aging/incubation test can give information on microbial transformation of arsenic [2,4] whereas long term column leaching tests simulates the in-situ leaching of contaminants in a field application of microbial immobilization [10]. Hence, there is a need to evaluate arsenic release in long-term tests that can predict arsenic leaching behaviour of such waste in anoxic as well as in oxic microbial environments.

With this aim present study reports on the stability of the biogenic sulphides (arsenic and iron sulphides) formed in an attached growth reactor (AGR) through a series of tests such as TCLP, aging and long

term leaching tests under different environmental conditions.

2. Materials and methods

2.1. Generation and collection of mixtures of reduced arsenic and iron bearing biosolids

An attached growth reactor (AGR) fabricated with perspex cylinder of 5 cm internal diameter (32 cm height and working volume 333 cm³), was inoculated with mixed bacterial culture. Seventeen cm of the AGR was initially filled with waste activated carbon (WAC) to support the growth of biofilm and used for treatment of arsenic and iron contaminated simulated and real groundwater [16]. Porosity of WAC bed was 0.432, which is equivalent to 144 cm³ of pore volume in the reactor bed. The AGR was backwashed under mixed flow of deoxygenated-deionized water and nitrogen gas at an interval of 4 days to remove the excess biomass as well as biosolids formed in it. The backwash suspension (BWS) was collected under anoxic conditions and used for batch aging test. The BWS were also centrifuged at 6000 rpm for 10 min to obtain wet paste, which was freeze dried and stored in sealed bottles in the –20 °C when not required. These freeze dried biosolids were used for mineralogical characterization and TCLP tests.

2.2. TCLP leaching tests

2.2.1. Estimation of arsenic and iron content in biosolids for TCLP test

One gram of dried biosolids was digested in a mixture of HNO₃ and HClO₄ (1:4, v/v) to extract out arsenic and iron from the solids [11]. After that digested samples cooled, the contents were diluted to 20 ml with DI water, mixed using a Vortex mixer and allowed to settle overnight at 4 °C. The supernatant was then carefully decanted and arsenic concentration in the digested extract was measured in atomic absorption spectrophotometer (SpectrAA 55B, Varian) equipped with a vapour generation assembly (VGA-77). Iron concentration was measured by phenanthroline colorimetric method. Average concentration of arsenic and iron in biosolids were calculated and expressed as mass (mg) of arsenic or iron per kg of dry solids.

The TCLP test was conducted in accordance with SW 846 test Method 1311 [17]. pH of the biosolids as measured as per method 9045d [18], falls in the low alkaline range (< 10). Therefore, 0.1 M glacial acetic acid and 0.064 M sodium hydroxide buffered at pH 4.93 ± 0.05 was selected as the extraction solution. The TCLP tests were conducted in Teflon screw cap bottles (test vessels) keeping a specific leachant-to-solid mass ratio (on wt/wt basis) of 20 in the mixture. Kinetic and extended TCLP tests were conducted either in nitrogen filled vessels (screw cap Teflon bottles) leaving 50% head space of the gas above the mixture or in vessels (beakers) exposed to air. Experiments in nitrogen filled vessels were conducted to take more accurate account for anoxic landfill conditions, whereas experiments in beakers were conducted to represent oxic condition. All experiments were carried out in triplicate to ensure the reproducibility. The mixture was agitated at 30 ± 2 rpm in end-over-end rotary shaker for 84 ± 1 h. The agitated mixture was centrifuged at 6000 rpm for 10 min, settled for 10 min. The supernatant was filtered through 0.45 µm filter paper, and the filtrate was analysed for soluble arsenic and iron. The following are the experiments conducted related to TCLP tests:

2.2.2. Kinetic TCLP leaching test

Kinetic TCLP leaching experiments were conducted in a test vessel

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