

ARSENIC REMOVAL FROM CONTAMINATED WATERS

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Abstract

Arsenic is a contaminant at 781 of 1,430 sites identified on the National Priorities List and in mining and mineral processing wastewaters, smelter wastes, and sites for manufacture of semiconductors, petroleum products, wood preservatives, animal feed additives, and herbicides. Arsenic affects ~4,100 municipal water systems nationwide and is difficult to treat to 10 ppb levels. Adsorptive media can remove up to 99% of arsenic from drinking water at costs for POU/POE applications of ~\$0.20/1,000gal.

Full-scale microbial arsenic removal/stabilization treatment costs of \$0.10/1,000 gal have been demonstrated for mining and ground waters to 2 ppb levels. Processes using magnetic activated carbon and bacteria/biopolymers; separately and combined (BIOMAC) have been demonstrated to treat high levels of Arsenic (V) to low levels under a wide range of water chemistry. BIOMAC benefits are expected to include other heavy metal removal, such as lead, copper, zinc, fluoride, selenium, and improvement in taste and odor.

Introduction

Arsenic remains one of the most difficult metals to remove from various waters to the low levels required under current National Primary Drinking Water Standards (NPDWS). The Environmental Protection Agency (EPA) has established a Maximum Contaminant Level (MCL) of 50 µg/L for arsenic in drinking water; this value will change to a 10 ppb national standard in 2006. The most common species of arsenic in oxygenated wastewaters is the pentavalent form arsenate (V). Arsenite (III) is the reduced trivalent form of arsenic and is the second most abundant species in oxygenated waters. Arsenic has been found in at least 781 of 1,430 National Priorities List sites identified by the Environmental Protection Agency and is one of the more common inorganic hazardous waste contaminants throughout the world. Mining, processing and refining of minerals, metals and most fossil fuels can release arsenic into the air and water.

The chemical characteristics of arsenic are dominated by the fact that it readily changes oxidation states or chemical form through chemical or biological reactions that are common in

the environment. Therefore, rather than solubility equilibrium controlling its mobility, it is usually controlled by redox conditions, pH, biological activity, and adsorption/desorption reactions (1). Inorganic arsenic exists primarily in the +3 or +5 oxidation states (depending on redox potential), and its reactions are influenced by pH, redox potential, dissolved organic or inorganic components, and colloids, especially Fe sulfides; Fe, Mn, and Al oxides and hydroxides; and organic matter. Arsenic (III) and (V) are bound, by ligand bonding mechanisms and arsenic (III) is readily precipitated as As_2S_3 or co-precipitated with FeS_2 or FeS . The aqueous solution chemistry of arsenic, relating to hydrometallurgical processes has been extensively covered in the literature, and the use of thermodynamic stability diagrams to describe the chemistry has been widely adopted.

Various methods considered to deal with arsenic in hydrometallurgical processes include: oxidation-reduction, precipitation and thermal (lower temperature precipitation – used in the semi-conductor industry) precipitation, co-precipitation, adsorption, electrolysis and cementation, solvent extraction, ion exchange, membrane separations including reverse osmosis, precipitate and ion flotation, and biological processing. Robins, Twidwell, and Young describe most of these methods in some detail (2-10). Precipitation, adsorption and cementation are possibly the processes that are presently being more generally adopted. The above methods for removing arsenic from process solutions and effluents have been used by the mineral process industries for many years. More recently, many of these technologies have been applied to treat arsenic in drinking water. However, with the requirement to reduce arsenic to a few parts per billion, the applicable technologies are limited. Low cost removal of arsenic from drinking water is likely to be confined to biological and/or combined biological-chemical precipitation, adsorption, or cementation (11-15).

Arsenic complexes that form in solution have received little attention; complexes of arsenic (V) with iron (III) are mostly studied (5,15). The removal of arsenic from gold process solutions has been of considerable interest over the years, and has been investigated more than other hydrometallurgical processes (5,16). There is much current interest in both *ex situ* and *in situ* stabilization technologies involving the use of microbial systems to immobilize contaminants present as anions under oxidizing conditions. Microbial reduction of arsenic and other metals and metalloids has been demonstrated in which anaerobic organisms reduce arsenic to insoluble forms including binding to iron and aluminum oxides, sulfide precipitates, and possibly elemental forms to remove arsenic to low levels ($<10 \mu\text{g/L}$), (17-22).

Activated carbon is produced from a variety of carbonaceous materials, including bones, coals, wood dust, peat, nutshells and wood charcoal through pyrolysis or carbonization of the raw materials. Activated carbon has surface groups that include mainly carboxyl, phenolic, quinone-type carbonyl, lactone, pyrone, carboxylic, and cyclic peroxide groups (23). Contact of activated carbon and aqueous solutions can be made using two main types of processes; contact-batch or semi-batch operation and column systems. A new magnetic activated carbon (MAC), developed at the University of Utah (24-26), can be easily separated from the solution using a magnetic separator; even those containing significant concentrations of solids.

Research goals are to develop combinations of inorganic, microbial, and biomaterial surface modifications to MAC that significantly increase arsenic removal or adsorption and to assess arsenic removal from mining, environmental, and drinking, waters. Benefits include new, cost effective arsenic remediation and treatment methods. Modified MAC materials – BIOMAC will simplify and improve arsenic treatment methods already in place for difficult-to-treat solutions.

Materials and Methods

Evaluation and characterization of arsenic removal must consider the variations in water chemistry of the vastly different waters that are being considered for treatment application. Thus, the materials and methods presented will be in accord with the current and recent testing conducted to investigate different components of the research goals presented above. At some point in this research, characterization will need to include arsenic availability, possible bioavailability, interferences caused by co-contaminants and different selected ions, site environmental conditions of pH, redox, temperature, dissolved oxygen, etc, and available carbon, nitrogen, phosphate, and sulfate concentrations. Additional potential limiting or interfering factors include, but are not limited to potassium, magnesium, calcium, manganese, and iron.

Arsenic Reducing Microbes

Generally, some combination of indigenous and augmented microbes has an advantage for both in situ and bioreactor applications and usually outperforms native microbes in most treatment situations. Arsenic reducing microorganisms were isolated from arsenic-contaminated mining process and waste waters and agricultural drainages throughout North America. Arsenic reducing bacteria tested individually and in biofilm populations included, but were not limited to *Alcaligenes*, *Escherichia*, *Pseudomonas*, *Desulfovibrio*, *Shewanella*, and *Bacillus sp.*, various sulfate reducing bacterial species. All were demonstrated to have high metal and other inorganic transformation kinetics under a variety of water chemistries.

Arsenic and sulfate reducing bacteria were used alone and in various combinations to test their ability to remove arsenic from drinking, environmental, and mining waters. All tests used a microbial support surface of activated carbon and special methods for conditioning the carbon prior to microbial biofilm establishment and growth. A combination of inorganic, organic, and microbial factors and methods are used in combination to induce establishment of a robust biofilm that was able to tolerate a broad range of pH and co-contaminant concentrations. The biofilms established can be controlled so that growth levels do not cause plugging of the bioreactor or in situ treatment. Activated carbon controls were included, without microbes, and experimental results subtract any arsenic adsorption by the treated carbon controls. Waters tested were at pilot-scale (55 gallon drum and flow rates up to 0.5 gpm) and included arsenic containing mining waste waters, environmental waters, and drinking water.

Magnetically Activated Carbon (MAC)

Synthetic aqueous solutions were used to determine arsenic adsorption by MAC. Analytical reagent grade chemicals including $\text{Na}_2\text{HAsO}_4 \cdot 7\text{H}_2\text{O}$ were used. All experiments used powdered MAC (100% <149 μm) obtained from pilot-plant scale production. A solution containing 10 mg/L arsenic at an initial pH of 4 was considered as the 'reference solution'. Solutions were contacted with 1g/L of MAC and were agitated at 200 RPM in a rotary shaker for 24 hours. Ten milliliter samples were taken at specific time intervals during the test to establish the kinetic curves. Variation in the initial arsenate solution pH, arsenic concentration, and MAC concentration, were tested and compared to results with the reference solution.

MAC impregnation tests were carried out using analytical reagent grade $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and $\text{ZrO}(\text{NO}_3)_2 \cdot x\text{H}_2\text{O}$ salts. Impregnation tests were done in agitated flasks (200 RPM) for 24 hours. The MAC concentration was maintained at 10g/L. The pH was not controlled but measured during the adsorption reaction. Impregnated MAC was used with the reference solution to compare its effectiveness for arsenic adsorption.

Results and Discussion

Figures 1 and 2 show the growth of various different microbial populations on activated carbon and subsequent arsenic removal using different nutrients or nutrient combinations. In these experiments, the treatment methods used for microbial biofilm establishment, generally blocks activated carbon arsenic adsorption. Therefore, what is observed is arsenic removal by a particular microbe or microbial population on an activated carbon material.

Figure 1 shows arsenic reduction in a 24 hr laboratory screening using artificial waters supplemented with 10 mg/L arsenic (V). As might be expected more complete nutrients, those at the back of the graph, stimulated a greater portion of the microbes and microbial populations and resulted in better arsenic reduction. Figure 2 shows a similar screening carried out with the more complete nutrients using site waters containing 3.5 mg/L arsenic as arsenic (V); retention time was 8 hrs. Some similar relationships can be seen with site waters and some of the same microbes and microbial populations as influenced by site water chemistry.

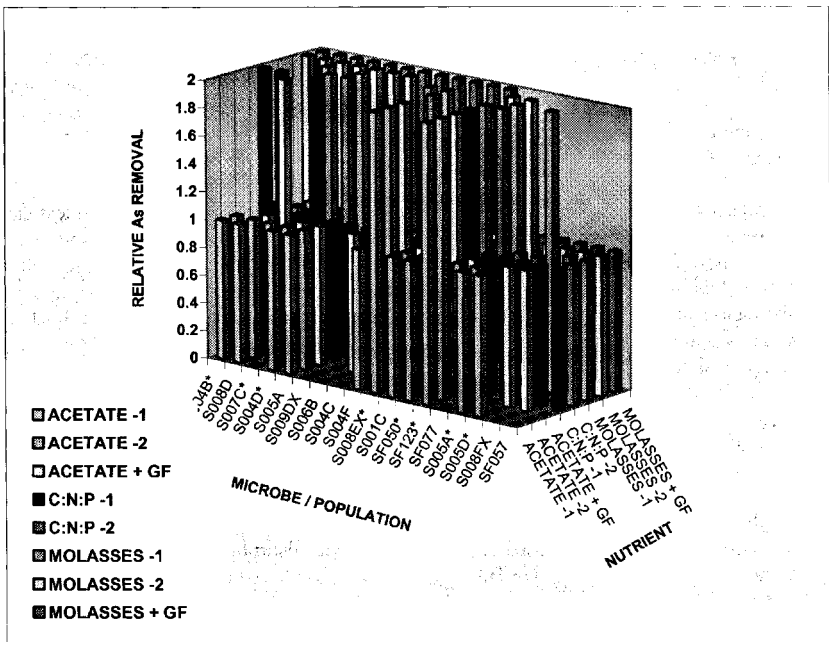


Figure 1. Relative arsenic reduction by various microbes and microbial populations (*). Lower concentrations of nutrient are denoted by (1), higher nutrient concentrations by (2), and richer or more complete nutrients by (+GF).

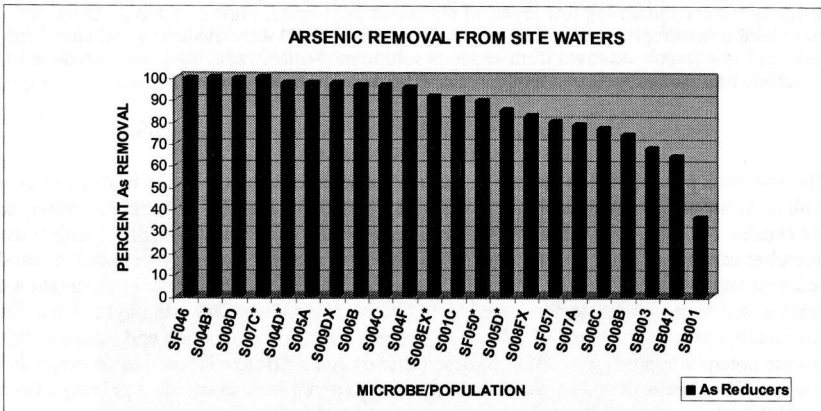


Figure 2. Arsenic reduction by microbes and microbial populations (*) using an enriched molasses-based mixture. Actual site waters contained ~3.5 mg/L arsenic as arsenic (V).

Arsenic Binding Biomaterials

Arsenic binding biomaterials were tested alone in tests examining arsenic complexation in solution, and bound to activated carbon. Tests included microbial metal binding components, biomaterials from waste food products, and protein mixtures. Results shown include arsenic adsorption to activated carbon and arsenic adsorption to activated carbon treated with a selected biomaterials complex shown to have a high arsenic removal in the waters tested. Two 4.5" x 1.0" polypropylene columns were filled with either activated carbon alone or activated carbon treated with biomaterials capable of absorbing arsenic. Over 400 bed volumes were processed before effluent arsenic was detected in the arsenic biomaterial column consisting of inorganics, microbial proteins, and enzymes. The carbon/ bacterial system was compared to the sorption capacities of activated carbon alone (both referenced and measured). The biomaterial and carbon laboratory data are presented in Figure 3.

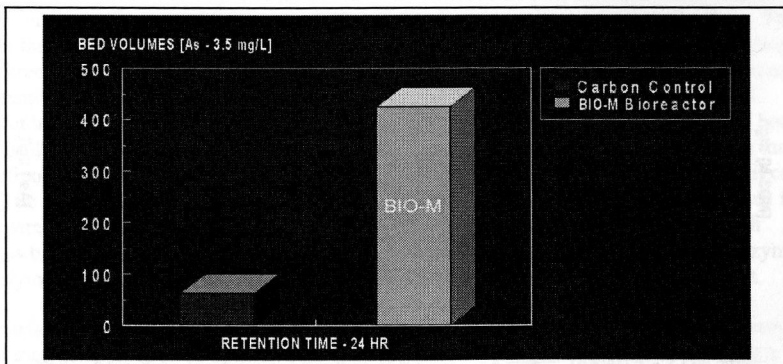
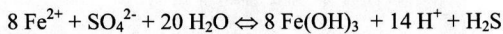


Figure 3. Arsenic adsorption by activated carbon alone and by activated carbon treated with biomaterials capable of adsorbing arsenic.

Live microbial populations were used to treat mining waters containing arsenic and nitrate and drinking waters containing low levels of arsenic at pilot-scale, Figures 4 and 5. Different combined heterotrophic and sulfate-reducing bacteria (SRB) were evaluated and later used in field tests for arsenic removal from aqueous solutions. Sulfate reducing bacteria reduce sulfate to sulfide as a part of their metabolic cycle. The reaction is as follows:



The arsenic is precipitated as arsenic sulfides. Arsenic sulfide precipitation is also carried out with heterotrophic microbes at lower levels and slower kinetics. Heterotrophic microbes also are capable of direct arsenic reduction and can reduce arsenic (V) to arsenic (III) with some microbes capable of reducing arsenic (III) to elemental arsenic. Arsenic precipitation could be accomplished with hydrogen sulfide gas. However, data from laboratory experimentation using arsenic containing waters demonstrated that effective arsenic removal using hydrogen sulfide gas requires pH's in the range of 2.5 to 3.0. Hydrogen sulfide production and subsequent arsenic precipitation using a combined heterotrophic and SRB population was accomplished without pH adjustment and has been demonstrated to precipitate arsenic in a pH range from 5.5 to pH 9.4 when established as a biofilm on activated carbon.

Two test series consisting of three [208 L (55 gal)] polyethylene test barrels, containing cylindrical pellets $\frac{1}{2}$ " by $\frac{1}{4}$ " of activated carbon as a microbial support surface, were used to remove arsenic. The inoculum was a consortium containing a combination of indigenous and inoculated microorganisms consisting of heterotrophic and SRB from several different sources. The microbial populations were screened for optimal arsenic and nitrate removal and fingerprinted to identify the microbes present. Bioreactors had a free volume of ~55% and were inoculated one week prior to set-up in the field. One test series treated mining wastewaters containing arsenic and nitrate and the other was developed to treat drinking waters containing arsenic. The mining wastewater contained ~0.65 mg/L As and ~100 mg/L nitrate nitrogen. The feed solution was amended with 20 mg/L ferrous sulfate heptahydrate and a molasses based nutrient mixture containing phosphate. The test lasted 130 days using bioreactor retention time of ~8 hours and produced arsenic effluents at near detection (<2 ppb) and nitrate levels near 0 mg/L (Figure 4).

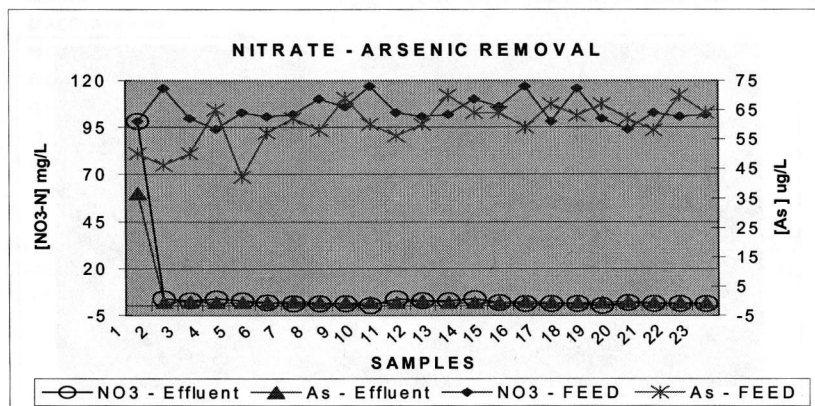


Figure 4. Arsenic and nitrate removal from mining waters at pH 6.3 using a combined heterotrophic microbial population in the same bioreactor series and a retention time of ~8 hrs.

The second test series was used to treat drinking water. This pilot-scale test was independently evaluated for microorganisms and nutrients required. An enriched molasses based nutrient and a similar, but not identical microbial consortium was used for this system. Arsenic in the effluent was consistently near or below detection (<2 ppb). Results are presented in Figure 5.

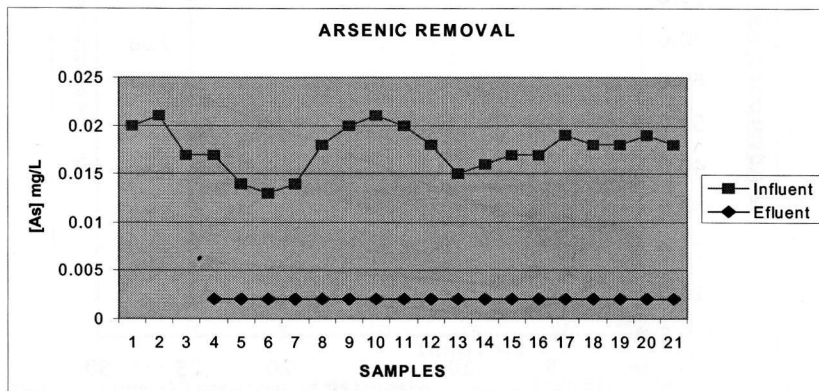


Figure 5. Drinking water treatment for arsenic removal at pH 6.5 using a combined heterotrophic and SRB microbial population and a retention time of ~8 hrs.

BIOMAC Testing

Activated carbon is used extensively for the removal of organic materials for water and wastewater treatment. Granular activated carbon is the best broad-spectrum technology for the treatment of both synthetic organic chemicals and natural organic material and is recommended by the EPA as one of the best available technologies for water and wastewater treatment (27,28). Under certain circumstances, activated carbon can also be successfully applied for the removal of heavy metals, such as As, Cd, Cr, Hg, Cu, Fe, V, Zn and Ni, particularly when present at trace levels (29-32). The proposed research is focused on the removal of hazardous metal ions and complexes from contaminated waters by adsorption on activated carbon. The aim of this work is to use conventional granular activated carbon (GAC) and newly developed powdered magnetic activated carbons (MAC) that have been modified in their surface structure by chemical/heat treatments to adsorb cationic species in solution. This innovative MAC product has two major advantages over comparable GAC products. First, MAC's small particle size results in higher metal-ion/complex loading capacity and higher adsorption kinetics than that of conventional granular activated carbon. Second, because the MAC is magnetic, it can easily be recovered by magnetic separation from streams to which it has been added, even from those streams that contain solids. Future testing will include combinations of MAC with various biomaterials including microbes, microbial components such as proteins and enzymes, biopolymers, and proteins from selected food processing waste into a BIOMAC product.

The surface functionality of the carbon plays an important role in its ability to uptake heavy metal ions/complexes and binding of biomaterials. Preliminary studies were undertaken to investigate the effect of MAC surface modification on the arsenic uptake ability. Figures 6 through 9 show the results of these tests.

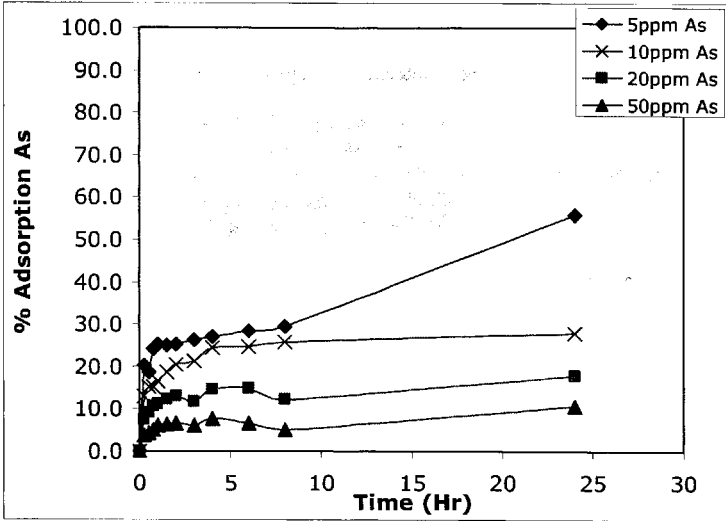


Figure 6. Arsenic (V) adsorption by magnetic activated carbon at different arsenic concentrations. Initial pH= 4.0; [MAC]= 1g/L; T=25°C; agitation= 200 RPM.

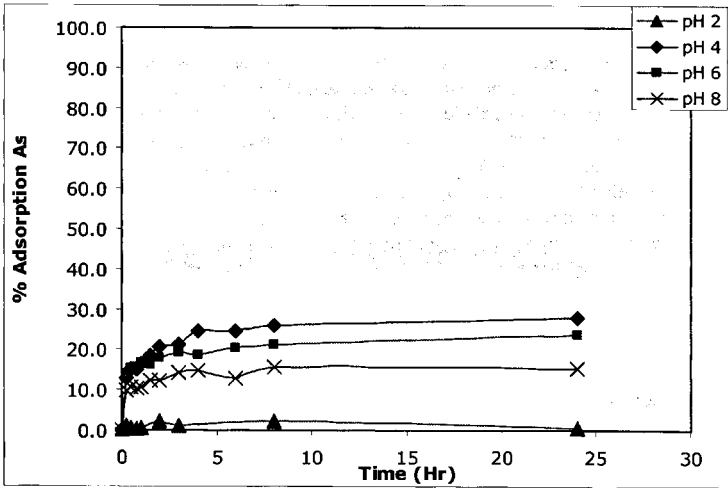


Figure 7. Arsenic (V) adsorption as a function of initial pH. [As]=10mg/L; [MAC]=1g/L T= 25°C; agitation= 200RPM

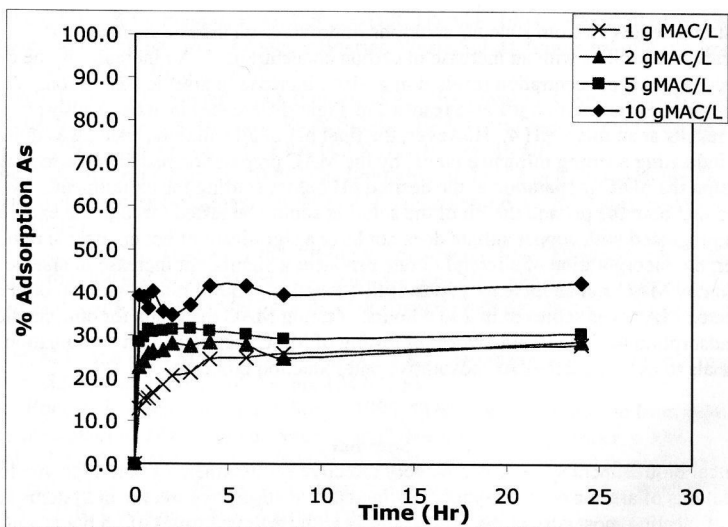
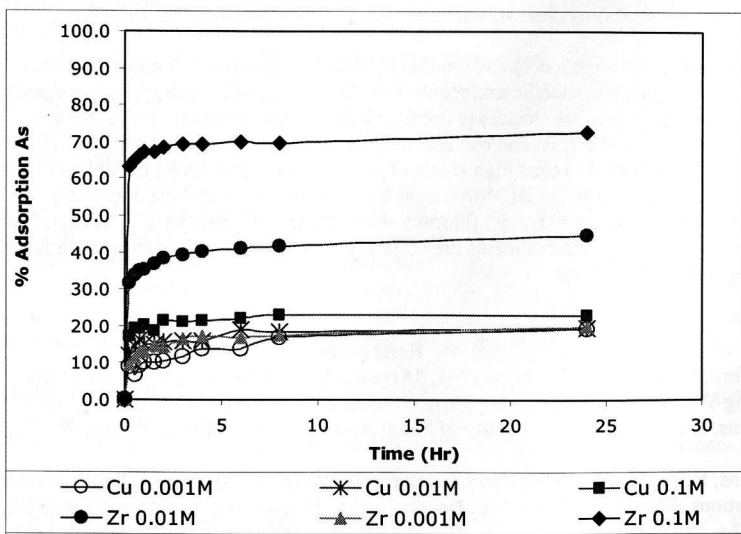


Figure 8. Arsenic (V) adsorption by MAC as a function of MAC concentration in solution. [As]=10mg/L; initial pH=4.0; T=25°C; agitation = 200RPM



Figures 9. Arsenic (V) removal using MAC pre-treated with different concentrations of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and $\text{ZrO}(\text{NO}_3)_2 \cdot x\text{H}_2\text{O}$ salts. [As]=10mg/L; [I-MAC]=1g/L; initial pH=4.0; T=25°C; agitation=200RPM

Removal of arsenic (V) from aqueous solutions improves with a decrease in arsenic concentration as well as with an increase in carbon concentration. An increase of one order of magnitude in MAC concentration resulted in a ~10% increase in arsenic adsorption. Variation of the initial arsenate solution pH also resulted in slight differences in arsenic adsorption, with the best results at an initial pH 4. However, the final pH of all solutions tested was quite similar, indicating a strong influence on pH by the MAC preparation tested and a requirement to condition the MAC preparation at the desired pH before starting the experiment. This also indicated that both the pH and the Eh of the solution should be tested through the experiments. MAC impregnated with copper sulfate does not have a significant effect on arsenic removal. However, the incorporation of zirconyl nitrate produces a significant increase in arsenic adsorption by MAC and an increase in adsorption kinetics. Equilibration with zirconyl nitrate impregnated MAC was achieved in 2 to 4 hours. Testing MAC materials for optimization of arsenic adsorption lays the ground work for testing of MAC materials with combination of biomaterials to examine BIOMAC adsorptive and reduction processes.

Summary

Economical biotreatment systems can be very effective for treatment of both high and low concentrations of arsenic contamination. Biological and adsorptive treatment systems are capable of meeting most current discharge criteria with treatment times of ~8 hours and costs ranging from ~\$0.04/1,000gal to ~\$0.55/1,000gal. Biological and adsorptive treatments are being designed and tested that are expected to meet current and planned drinking and aquatic wildlife criteria; arsenic can be removed to below detection. Microbial precipitates produced are also thousands of times less voluminous than conventional chemical treatments.

Biological and adsorptive media and combined BIOMAC treatment of mining waters is expected to be quite site specific and requires evaluation of site chemistry, microorganisms, and other key environmental parameters to optimize contaminant removal. Processes using magnetically activated carbon and bacteria/biopolymers, separately and combined (BIOMAC), have been demonstrated to treat high levels of Arsenic (V) to low levels under wide water chemistry ranges. Additional BIOMAC benefits are expected to include other heavy metal removal, such as lead, copper, zinc, fluoride, selenium, and improvement in taste and odor. Some full-scale arsenic biotreatments are currently available for review along with factors effecting biotreatment costs.

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