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USING ALGAE TO CLEAN UP WATER

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INTRODUCTION

The use of microorganisms in the treatment of hazardous wastes containing both inorganic and organic pollutants is becoming more and more common. There have been two approaches to the use of microorganisms in waste treatment. One involves the use of living organisms and the other involves the use of nonviable biomass derived from microorganisms. While the use of living organisms is often successful in the treatment of toxic organic contaminants, living organisms have not been found to be useful in the treatment of solutions containing heavy metal ions. This is because once the metal ion concentration becomes too high or sufficient metal ions are adsorbed by the microorganism, metabolism is disrupted causing the organism to die. disadvantage is not encountered if nonliving organisms or biological materials derived from microorganisms are used to adsorb metal ions from solution. Instead, the biomass is treated as another reagent, a surrogate ion exchange resin. The binding, or biosorption, of metal ions by the biomass results from coordination of the metal ions to various functional groups in or on the cell. These chelating groups, contributed by the cell biopolymers, include carboxyl, imidazole, sulfhydryl, amino, phosphate, sulfate, thioether, phenol, carbonyl, amide and hydroxyl moieties.¹

Various algal species and cell preparations have quite different affinities for different metal ions.²³ The different and unusual metal binding properties exhibited by different algae species are explained by the fact that various genera of algae have different cell wall compositions. Thus, certain algal species may be much more effective and selective than others for removing particular metal ions from aqueous solution.⁴

The reaction of metal ions with a nonliving algal cell is depicted in Figure 1. This reaction shows the interaction of divalent or trivalent metal ions with either a living or nonliving algal cell to form a complex composed of the algal cell and the metal ions. The result of this reaction, that is, the formation of the alga-metal ion complex is basically why metal ions are toxic to living organisms and shows how the toxic effect of metal ions is amplified in the food chain. The metal ions are adsorbed to the cell even when their concentrations are in the mg/L range. The bound metal ions, when accumu-

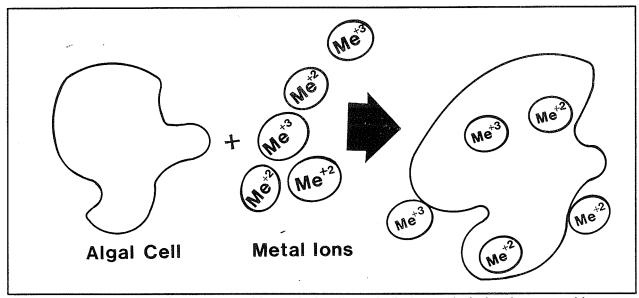


Figure 1. The Reaction of Metal Ions With an Algal Cell. Both divalent and trivalent heavy metal ions react with chemical groups on an algal cell wall to form an alga-metal ion complex.

lated over time, eventually interfere with metabolism by disrupting enzyme reactions and killing the organism. If microorganisms on which metal ions have been sorbed are used as a food source by larger organisms, the metal ions find their way into the food chain. This can eventually result in toxic effects for humans.

While the reaction shown in Figure 1 has been known for many years, it is only recently that advantage has been taken of the high affinity of microorganism cell walls to remove and recover metal ions from industrial waste water or contaminated ground waters. Methods to reverse the reaction shown in Figure 1 have been developed so that when metal ions are recovered from dilute solutions, they can be stripped off the cell walls in a highly concentrated form. The cells can then be reused to capture more metal ions from dilute solutions. Conditions can also be adjusted so that only one or two types of metal ions can be sorbed from solution and then they can be selectively stripped from the algal cell one metal at a time.^{1,5}

AlgaSORB®: A NEW BIOSORBENT FOR WASTE-WATER TREATMENT

Bio-recovery Systems, Inc. has developed a proprietary, algal based material, AlgaSORB®, which can be used on a commercial basis to remove and recover heavy metal ions from point-source

industrial waste water, contaminated ground waters or mining process streams. AlgaSORB® functions very much like a commercial ion exchange resin. It can be packed into columns through which waters containing heavy metal ions are flushed. The heavy metal ions are adsorbed to AlgaSORB® and metalfree water exits the column for reuse or discharge. Once the AlgaSORB® is saturated with metal ions, the metals can be stripped from the AlgaSORB®, which is then ready for reuse. In comparison to ion exchange resins, however, AlgaSORB® has some distinct advantages making it superior to ion exchange resins for certain applications. In other instances, ion exchange resins perform better than AlgaSORB®. AlgaSORB® has a remarkable affinity for heavy metal ions; in some cases the metalbinding capacity is as much as 10 percent of the dry weight of the cells. The algae matrix is capable of concentrating heavy metal ions by a factor of many thousandfold.

When unadulterated algal cells are packed into columns, the cells tend to aggregate and form cohesive clumps through which it is difficult to force water even under high pressures. However, when the cells are immobilized into a polymeric matrix, this difficulty is alleviated.

The algae are killed in the immobilization process indicating that sorption does not require a living organism, and hence the algal matrix can be exposed, with little or no ill effects, to solution conditions that would normally kill living cells. The pores of the polymer are large enough to allow free diffusion of ions to the algal cells, since similar quantities—of metal ions are bound by free and immobilized cells. The immobilization process serves two purposes: (1) It protects the alga cells from decomposition by other microorganisms. AlgaSORB® immersed in aqueous solution for over two years has shown no decrease in metal binding efficiency. (2) It produces a hard material that can be nicely packed into chromatographic columns that can be pressurized and that have excellent flow characteristics.

Not only does the immobilized algal matrix appear to be useful for the removal of the "traditional" heavy metals from solution, but it also seems to be particularly useful for near quantitative removal and recovery of very low concentrations (in the parts per billion range) of precious metals such as gold, silver, platinum and palladium. In addition, conditions have been developed for removing metallic oxoanions such as chromate from aqueous solutions using immobilized algae.

AlgaSORB® functions as a "biological" ion exchange resin and like ion-exchange resins, can be recycled. Metal ions have sorbed and stripped over many cycles with no noticeable loss in efficiency. In contrast to current ion exchange technology, however, a real advantage of the algal matrix is that the components of hard water (Ca⁺² and Mg⁺²) or monovalent cations (Na⁺ and K⁺) do not significantly interfere with the binding of toxic, heavy metal ions. In fact, calcium or magnesium ion concentrations as high as 10,000 mg/L have little or no effect on AlgaSORB® sorption of copper at concentrations as low as 6.5 mg/L. The binding of Ca⁺² and MG⁺² to ion-exchange resins (even chelating ion exchange resins that are relatively selective for transition metal ions) often limits ion exchange usefulness since calcium and magnesium ions are frequently present in high concentrations and compete for heavy metal ion binding. This means that frequent regeneration of ion-exchange resins is necessary to remove effectively heavy metal ions from solutions. It also means most of the ion exchange capacity is spent in removing calcium and magnesium from the waters. Thus, AlgaSORB® has the potential to be particularly useful for removing metal ions from "hard" waters.

AlgaSORB® is also particularly effective for heavy metal removal from waters containing organic residues. Organics often foul synthetic ion exchange resins that limits their utility in many waste-water

treatment applications, including ground-water treatments. AlgaSORB[®], on the other hand, functions well in waters containing organic molecules.

WASTE STREAMS FOR WHICH THE AlgaSORB® TECHNOLOGY IS APPLICABLE

A. Industrial Point Sources

A major source of heavy metal wastes from industrial sources comes from the electroplating, metal finishing and printed circuit board manufacturing industries. Waste waters from these industries primarily come from rinsing operations. The rinse waters will typically contain rather low concentrations (on the order of 100 parts per million) of heavy metal ions. Certain of these waste streams are particularly amenable to treatment with AlgaSORB. The metal can be recovered and then either recycled back into the process or recovered for use by other industries.

B. Ground Waters and Surface Leachates

Contaminated ground waters and surface leachates often contain heavy metals in the low parts per million or even parts per billion range. The AlgaSORB® technology is particularly well suited for removing and recovering heavy metal ions from these waters, which will often contain high concentrations of nontoxic dissolved materials. Often these types of waters will contain high concentrations of sodium, potassium, magnesium, chloride or sulfate that are innocuous and for which no treatment is needed. The AlgaSORB® is capable of preferentially removing heavy metals found in these streams. Toxic heavy metal ions recoverable with the algal biomass include copper, nickel, uranium, lead, mercury, cadmium, zinc, arsenic, and silver among others.

C. Drinking Water

In locales with older plumbing, heavy metals such as lead are often found in tap waters. Because AlgaSORB® has such a high affinity for heavy metal ions, ions such as lead can be removed with AlgaSORB® to levels well below those allowed in drinking water.

D. Precious Metal Recovery

AlgaSORB® has a higher affinity for precious metal ions than other heavy metal ions tested. 5.6 Thus, another area in which the AlgaSORB® technology is useful is in the recovery of gold, silver, or platinum group metals from mining process streams, waste waters resulting from mining operations, and industrial point source waste water.

ADVANTAGES OVER CONVENTIONAL WASTE TREATMENT

A. Disadvantages of Conventional Treatment

Sludge Disposal Costs

The conventional method for treating waste waters in electroplating or printed circuit board manufacturing plants has been to commingle all metal-containing waste waters and send them to a central location for treatment. Treatment methods vary depending upon what metals are present in the stream, but the most common treatment is precipitation of the metals as hydroxides. If metal cyanide complexes are present, cyanide is usually oxidized prior to metal precipitation. Likewise, if hexavalent chromium is present, it is usually reduced to trivalent chromium prior to precipitation. The metals hydroxide precipitates are then dewatered and most commonly sent to a hazardous waste landfill. As of 8 August 1988, these metal-containing sludges could no longer be sent to a hazardous waste landfill unless they are stabilized so that the toxic metal ions cannot be leached from the sludge. A variety of agents such as Portland cement fly ash or other pozzolanic materials can be used to stabilize the sludge. However, whatever the stabilization method, the disposal costs have increased dramatically since August 1988. In addition, both state and federal regulatory agencies are moving toward the future complete ban of land disposal of metal hydroxide sludges in any form.

2. Difficulty of Cost-Effective Treatment

In addition to high sludge disposal costs, another disadvantage of the conventional treatment system is the difficulty in many instances of reaching effluent metal conditions low enough to meet discharge standards. This is because hard-to-treat waters are often commingled with easy-to-treat waters thereby making all the waste water hard-totreat. For example, in printed circuit board manufacturing operations, there are typically three different types of copper-bearing waste waters that must be treated: copper sulfate from acid copper baths, ammoniacal copper from alkaline etchers and chelated (usually EDTA quadrol or tartrate) copper from electroless copper baths. Copper sulfate responds very well to hydroxide precipitation, but the ammonia complex of copper and the EDTA chelate of copper are very difficult to treat with conventional hydroxide precipitation. Thus, expensive chemicals such as sodium borohydride or dithiocarbamates are added to the entire waste-water stream in order to treat the ammoniacal and chelated copper, which usually make up only a small proportion of the total waste streams.

3. High Water Consumption

When the conventional hydroxide precipitation of metals is used, usually sodium hydroxide or lime along with other reducing agents or flocculating agents are added to produce the metal hydroxide sludge. Once the sludge is removed from the waste water, the water is generally discharged to a sewer. No opportunity exists for reuse or even partial reuse of the water because of the effluent water has too many dissolved salts to be effective as a rinse water. The cost of deionizing this water is generally much higher than the cost of deionizing fresh tap water and hence water reuse is generally not a viable economic option.

4. <u>Liability for Sludge Disposal</u>

Generators of toxic metal sludges are held liable, without proof of fault, for cleaning costs and natural resource damage at hazardous waste disposal sites at which the generator's waste is disposed. Therefore, if the owners of a hazardous waste dump happen to mismanage the site so that toxics are allowed into the environment, it is the generator who is ultimately responsible for the cleanup. Thus, any process by which sludge can be minimized or eliminated will reduce liability for the generator.

B. Advantages of a Recovery-Recycle Approach to Waste Treatment

The AlgaSORB® technology has been incorporated into a highly effective recovery-recycle approach to waste-water treatment for the electroplating, metal finishing, and electronics industries. The concept is illustrated in Figure 2 for a treatment system that allows for recovery of metals and recycling of process waters. In this scheme, rinse waters derived from each individual plating bath are segregated and passed through columns containing AlgaSORB® or specialty ion exchange resins. Metal ions are removed from the rinse waters, which can then be discharged directly or returned to the rinse tanks for partial water reuse. Because salts tend to build up in the rinse waters, deionization of the treatment effluent may be needed if it is to be reused in crucial rinses. Otherwise, a bleed-off of water to the sewer is adequate to keep salt-build up at acceptable levels. Such an approach can often decrease water usage by 50 to 90 percent.

Once the columns of AlgaSORB® are saturated with metals, the metal ions can be stripped from the columns. The concentration of the stripped metals is approximately 10 g/L. In certain instances, these stripped metal ions can be added back to

Using Algae to Clean Up Water RECYCLE / RECOVERY SYSTEM

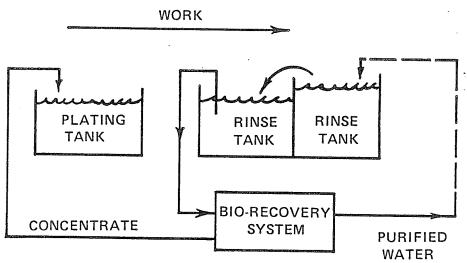


Figure 2. Recycle-Recovery System. Segregated rinse waters from a plating process are directed through a recovery system where metal ions are recovered, and the rinse waters are directed back to the rinse tanks.

the plating bath. In instances where this is not acceptable, the metal can be recovered through electrowinning or metalwinning. Alternatively, the metal ions can be further concentrated by evaporation and sent to one of a number of companies now established to recycle such materials. Whichever approach is taken, however, the elimination of sludge production results in lower operational costs due to decrease in chemical costs, decrease in water usage, elimination of sludge disposal costs, and minimization of future liability.

STATE OF DEVELOPMENT

Bio-recovery Systems is currently manufacturing and installing waste-water treatment systems for use in recovering heavy metals from industrial point sources in the electroplating and printed circuit board manufacturing industries. Figure 3 shows a system designed for a printed circuit board manufacturer. The heart of the system is comprised of columns (B), which contain the metal-adsorbing materials. Rinse waters containing only a single type of plating or etching chemistry are segregated and plumbed to individual columns. When the columns become saturated with metal ions, a specific metal ion sensor signals the controller (A) to begin a regeneration cycle to strip the metals from the materials in the column and to send the stripped metal ions to one of the holding tanks (D). Once regeneration is complete, the controller automatically returns the regenerated column back into service. The stripped metals are then recovered as the metallic elements in the metalwinning unit (E).

The system shown in Figure 3 is capable of treating 30 L/min (8 gal/min). However, larger flow rates (up to hundreds of gallons per minute) can be accommodated by simply adding either more metal-adsorbing columns or by using larger diameter columns.

The system shown in Figure 3 was designed for a printed circuit board manufacturer, but the same type of system is also employed for metal finishing and electroplating facilities. Different chemistries are encountered in metal finishing rinse waters, but the approach to treatment of these waters is basically the same as that encountered in a printed circuit board manufacturer's facility, that is, waste waters are segregated for treatment so that maximum reuse of metals and water can occur.

AlgaSORB® PERFORMANCE EVALUATIONS

In 1986 and 1987, Bio-recovery Systems, Inc. was awarded Small Business Innovative Research (SBIR) contracts from the United States Environmental Protection Agency to research and develop the AlgaSORB® technology for commercial applications. In 1988, Bio-recovery Systems was awarded another EPA contract as part of the Emerging Technologies program under the auspices of the Superfund Innovative Technologies Evaluation

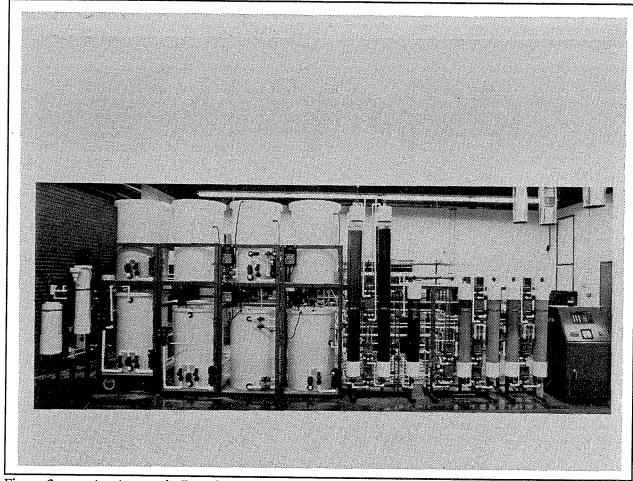


Figure 3. An Automatic Recycle-Recovery Waste Water Treatment System. A: controller; B: metal adsorbing modules; C: deionized water system; D: holding tanks for pH adjustment, regenerant chemicals; E: metalwinning module.

(SITE) program. Results from these contracts, some of which are summarized below, clearly show the efficiency of AlgaSORB® for heavy metal removal from a variety of sources. These successful laboratory and pilot scale tests made full scale commercialization of the technology a foregone conclusion.

A. Removal of Cadmium from Waters at a Superfund Site

Officials from EPA Region II arranged to supply samples from a well at a Superfund site in New Jersey, the Waldick Aerospace Devices site. These waters contained, among other things, cadmium at a level of 0.13 mg/L. The waters at a pH of 6.0-7.1 also contained, among other organics, 0.66 mg/L of a halogenated hydrocarbon, tetrachlor-

oethylene. Organics, of course, are well known to interfere with the function of traditional ion exchange resins.

A column containing AlgaSORB® (0.7 cm i.d. x 13 cm high) was prepared and the Waldick Aerospace waters were passed through the column. Five ml fractions of water exiting the column were collected until 500 mL (100-bed volumes) of Waldick waters were passed through the column at a flow rate of one-sixth of a bed volume per minute (total bed volume was 5.0 ml). Each fraction of effluent was analyzed for cadmium using graphite furnace atomic absorption spectrometry. All effluent fractions showed that cadmium concentration was near or below 0.001 mg/L through the passage of the 100-bed volumes of the cadmium-containing solution. Because the experiment was stopped after the pas-

sage of 100-bed volumes through the column, it is not possible to state explicitly what volume of solution could be treated before cadmium breakthrough would occur. However, experience has shown that if a test material is capable of treating at least 100-bed volumes of metal-bearing water, use of that material is economically feasible. The essential point is that AlgaSORB® removed cadmium well below those levels allowed in drinking water. The current drinking water levels for cadmium stand at 0.005 mg/L.

After 100-bed volumes of the cadmium-containing solution had passed through the Alga-SORB®-containing column, cadmium was stripped from the column by passing 0.15M H₂SO₄ through the column. Analysis of the column effluents showed that nearly 90 percent of the cadmium was stripped from the column with the passage of two-bed volumes of sulfuric acid through the column. Most of the remainder of the cadmium appeared in the next two-bed volumes. Mass balance calculations showed that, within experimental error, all of the bound-cadmium was stripped from the column.

B. Removal of Copper from Contaminated Ground Waters Containing Halogenated Hydrocarbons

Bio-recovery Systems obtained ground waters contaminated with copper, tetrachloroethylene, and dichloroethylene by a printed circuit board manufacturer. These waters contained a total dissolved solid content (TDS) of nearly 2000 ppm and had a total calcium and magnesium content of approximately 300 ppm. Past experience had shown that ion exchange resins were not effective in treating these waters for copper removal because of (1) the high mineral content and (2) the propensity of the resins to become clogged with the organics in these waters. However, experiments showed that 400-bed volumes of the copper-containing waters could be passed through a column (0.7 cm i.d. x 13 cm high) containing AlgaSORB® without effluents from the column containing more than 0.01 ppm of copper. The experiments were stopped at 400-bed volumes, so undoubtedly, larger volumes of waters could have been treated before unacceptable levels of copper appeared in the effluents.

After 400-bed volumes had been passed through the AlgaSORB® column, the bound copper was, within experimental error, completely stripped from the column by passing 0.5M H₂SO₄ through the column. Again, as with the previously described cadmium stripping, the copper was almost complete-

ly stripped within the first few bed volumes of effluent.

C. Removal of Mercury from Contaminated Ground Waters

Bio-recovery Systems was provided with water samples from a mercury-contaminated ground-water site. The site had been contaminated with mercury years ago as a result of a process used to manufacture chlorine from seawater. The ground waters contained 2-3 ppm of mercury (both inorganic and organic mercury), had a total dissolved solid content of 7,200 mg/L, and contained over 900 mg/L of calcium and magnesium. Passage of these mercury-containing waters through an AlgaSORB® column (0.7 cm i.d. x 13 cm high) resulted in effluents containing mercury at levels below 0.006 mg/L as determined by analysis using cold vapor generation and atomic absorption spectrometry. The customer requires effluents of below 0.01 mg/L for discharge.

These experiments show, as had earlier experiments, that AlgaSORB® is effective in removing both inorganic and organic mercury from aqueous solutions even in the presence of very high concentrations of calcium, magnesium, and other dissolved salt.

D. Removal of Nickel and Chromium from a Contaminated Ground Water

A sample of ground water contaminated with nickel and chromium was obtained directly from the electroplating business responsible for the contamination. Currently, the electroplater is pumping and treating these ground waters with his conventional (precipitation) waste-water treatment system. The initial pH of the ground waters was near pH 7. The chromium content (essentially all hexavalent chromium) was near 0.9 mg/L and nickel content was at 2.7 mg/L. The customer's discharge levels are 0.5 mg/L and 0.25 mg/L for nickel and chromium, respectively.

Passage of these waters through a column (6 mL total volume) containing AlgaSORB® resulted in effluents that were below 0.5 ppm in nickel after elution of 175-bed volumes. The nickel could be easily stripped by passage of acid through the column. However, chromium appeared in the column effluents after the passage of only five-bed volumes of the metal-contaminated ground water through the column. This was actually what had been anticipated since other work has shown that chromium(VI) is most strongly bound to AlgaSORB® at pH 3.5 and is not bound at pH values near 7. Thus, after adjustment of the pH of another portion of these

waters to pH 3.0 and passage through another AlgaSORB® column, results showed that after elution of 225-bed volumes of chromium-containing waters through the column, chromium content in the effluent was near or below 0.3 mg/L. Thus, these waters are successfully treated using two Alga-SORB® columns, if the pH of the effluent from the first column is adjusted to pH 3 before passage through the second column.

E. Removal of Ammoniacal Copper from Industrial Waste Waters

Many printed circuit board manufacturers use an ammonium hydroxide/ammonium chloride etch solution to remove copper from printed circuit boards. The resulting ammoniacal copper solution is particularly difficult to treat with conventional

precipitation technology because the copper-ammonia complex is stable in even very alkaline solutions. Figure 4 shows results of AlgaSORB® removal and recovery of copper from an ammoniacal copper waste water from a printed circuit board manufacturer. The influent concentration of copper was 286 mg/L. This solution was passed through a column of AlgaSORB® which contained 0.25 g of algae. Breakthrough of copper began to occur after approximately 75 mL of solution had been flushed through the column. The amount of copper (21.5 mg) bound to the AlgaSORB® at breakthrough corresponds to an 8.6 percent loading (on a dry weight basis) of the algal material with copper. Figure 4 shows that once the column was saturated with copper, the copper was quantitatively stripped with 0.5M sulfuric acid.

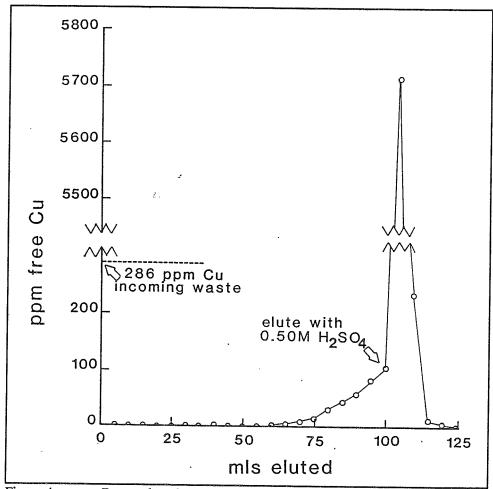


Figure 4. Removal and Recovery of Copper from an Ammoniacal Copper Solution. A waste solution containing copper as the ammonia complex was passed through a column containing AlgaSORB.

F. Removal of Lead from Printed Circuit Board Manufacturing Waste Waters

The printed circuit board industry frequently plates a tin-lead alloy onto printed circuit boards as a base for solder connections. The tin-lead alloy is plated from a solder bath, which often contains tin and lead fluoborates. Since tin discharge is notcurrently federally regulated, the major problem in treating rinse waters derived from tin-lead solder baths is lead removal. One particular AlgaSORB® preparation is especially amendable for this application since it strongly binds lead and allows the majority of the tin to pass through.

A sample of a tin-lead plating bath was obtained from a printed circuit board manufacturer. The bath rinse waters commonly contain 10-60 mg/L of lead and about twice as much tin.

A column containing AlgaSORB® (3.3 mL total bed volume) was prepared and the tin-lead containing waters (27.4 mg/L of lead; 49 mg/L of tin) which had first been adjusted to pH 5.0 were passed through the column at a flow rate of one-third of a bed volume per minute. Two-bed volume fractions of the effluent were collected, and each of these fractions was analyzed for tin and lead by atomic absorption techniques. All effluent fractions showed lead concentrations at or below the detection limit

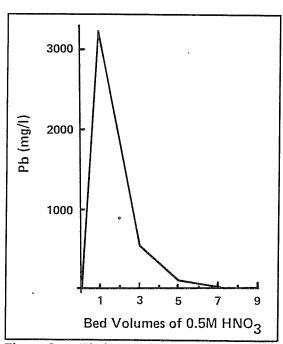


Figure 5. Elution Profile for Lead Stripping from AlgaSORB®.

of 0.1 mg/L for the first 300-bed volumes, after which lead began to appear in the effluents. Influent tin-lead passage was stopped after passage of 325-bed volumes through the column after which the column was stripped of lead by elution with 0.5M nitric acid (Figure 5).

All fractions eluted through the AlgaSORB® column were also analyzed for tin. Because tin is more weakly bound than lead, tin began to exit the column after passage of only 33-bed volumes of influent. Thus, the AlgaSORB® column showed marked preference for lead over tin. When the column was stripped of lead (after 325-bed volumes) the small amount of tin bound on the column was also fully recovered in the nitric acid stripping solution.

SUMMARY

The currently commercialized AlgaSORB® technology has been found to be particularly useful for treatment of various types of waste waters. Not only can waters be effectively treated, but the metals can be recovered and recycled. AlgaSORB® is a biological ion exchange material that has advantages over commercial ion exchange resins for particular applications.

In the past, the majority of waste waters containing heavy metals have been treated to produce sludges, which are then disposed in a landfill. It is becoming apparent that this approach is becoming less economical and that the federal regulations are aimed toward the complete elimination of landfill disposal of heavy metals in the future. Thus, the recycle-recovery approach for waste-water treatment will expand rapidly in the future.

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