CRADA FINAL REPORT [1]

CRADA No. Y1295-0356

with

CommonWealth Enterprises, Inc.

for

***In-Situ* Soil and Water Remediation at Contaminated Sites Utilizing a New Form of Humic Matter Called "Humosorb"**

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Abstract

This CRADA arose through the United States Industrial Coalition, Inc. to foster new business partnerships between the US and the Newly Independent States (NIS) of the Former Soviet Union under DOE's Incentives for Proliferation Prevention (IPP) Program. The main goal the DOE/IPP program is development commercially viable technology that will provide employment for NIS weapons specialists and diversion of equipment and facilities from weapons-related production. The NIS scientists involved in this CRADA had developed-and sought assistance in commercializing-a product called "Humosorb". The goal of this project, which involve collaborative work between a US company, the Oak Ridge National Laboratory (ORNL), and Russian weapons scientists at the Scientific Institute for Chemical Means of Plant Protection (SICPP), was to evaluate and field validate the application of Humosorb for in-situ remediation of metal-contaminated soils, specifically by immobilizing heavy metals in contaminated soil and preventing their uptake into plants.

Problems were encountered in fully accomplishing the goals and objectives of the CRADA. The US company, CommonWealth Enterprises (CWE), failed to fulfill their commitments, and the CRADA was unilaterally terminated by ORNL for nonperformance. Because of the promising results by the NIS scientists and ORNL, DOE continued funding through the CRADA' s full two year performance period.

Significant technical progress was made in testing and validating the use of Humosorb in metal remediation. The product was fully characterized with respect to its chemical and physical properties. The extent of immobilization of eight heavy metals to Humosorb was determined and the effect of complex mixtures of toxic metals was also examined. Greenhouse studies demonstrated that Humosorb causes very large reductions in the bioaccumulation of metals by plants and results in a corresponding decrease in plant toxicity .In a field demonstration experiment in Moscow, the mobility of heavy metals in soil was decreased by application of Humosorb, and the protective effects to plants observed in the greenhouse studies were also observed at the field scale.

In spite of the problems with the CWE, the principal objectives of this DOE/IPP Program have been achieved as a result of this CRADA. Another US company was recently formed to commercialize Humosorb for environmental remediation in the US. This company, Stable Earth Technologies, has formed a joint venture with the former

Russian weapons scientists, and are beginning to apply Humosorb (which they have re-named "Stabilite") in remediation of mercury-contaminated soil. Further commercialization activities are being pursued that will likely lead to new projects and areas of research in this direction. In particular, the use of Humosorb/Stabilite to remediate soil contaminated with polychlorinated biphenyls will be pursued, possibly within the context of another IPP CRADA. In addition to commercialization within the US, the NIS weapons scientists have been able to achieve commercial success in Russia. Financial support from this CRADA, as well as technical results developed by SICPP and ORNL during the course of this CRADA contributed to the commercial success of these former weapons scientists.

Statement of Objectives

This CRADA arose through the United States Industrial Coalition, Inc. to foster new business partnerships between the United States (US) and the Newly Independent States of the Former Soviet Union (NIS) under the U.S. Department of Energy (DOE) Incentives for Proliferation Prevention (IPP) Program. The

CRADA involved the US business, CommonWealth Enterprises, Inc. (CWE), the Oak Ridge National Laboratory (ORNL), and the Scientific Institute for Chemical Means of Plant Protection (SICPP) in Russia. The NIS scientists had developed-and now sought assistance in commercializing-a process for efficient and cost-effective extraction of humic acids from brown coal. The resulting product was called "Humosorb". Humic materials possess properties that make it potentially attractive for a variety of uses, as outlined in the description for Humosorb provided in the proposal sent to the US Industrial Coalition by CommonWealth Enterprises. Information supplied by the technology's developer and based on the known properties of humic substances suggests that Humosorb may be very useful in some applications in some environments, including immobilization of toxic metals and radionuclides in soil, as well as reducing the uptake of the metals by plants. This application of Humosorb would be of great value in the US and in foreign markets as a cost-effective solution to in-place soil contaminants. However, humic material is very complex and variable, and it properties and behavior cannot be readily extrapolated from one environment or application to another. Information was needed to develop a capability to predict the behavior of the product over a range of applications under a variety of site-specific conditions. Work identified in this CRADA seeks to develop understanding of Humosorb properties and behavior from its current status as an anecdotal and empirical set of site-specific observations, to a reliable and predictable understanding based on descriptions of basic properties and activities of the product. The focus of this CRADA was limited to application of Humosorb to remediation of metal contaminated soil. The remedial effects of Humosorb were postulated to arise from it's ability to stabilize metals in soil, preventing their leaching to groundwater and uptake by plants.

The goal of this project was to evaluate and field validate the application of Humosorb to immobilize heavy metals in contaminated soil and prevent their uptake into plants. Specific objectives included evaluating the benefits and limitations of Humosorb in small-scale experimental systems; confirming the immobilization of toxic metals and reduced metal uptake by plants in greenhouse studies; and demonstrating the effectiveness and feasibility of Humosorb in several experimental field applications. Three principal tasks were identified to achieve these objectives. The organization of the tasks reflects a progression: (a) well-controlled laboratory studies that test chemical interaction of Humosorb with metals and with the soil, (b) larger-scale greenhouse studies conducted over longer time-scales that include monitoring of uptake of metals into plants, and finally (c) several long-term field studies at metal- contaminated sites in Russia and the United States. Briefly the tasks included:
TASK 1: *Laboratory Studies on Interactions of Humosorb with Heavy Metals and with Soils* -This task examined both the binding of toxic metals to Humosorb as well as the adsorption of Humosorb-metal complexes to soils under a range of environmentally-relevant conditions. The objectives of the task were to determine the appropriate application rates for Humosorb to promote binding of toxic metals to soils. Most of the laboratory work was conducted by the Russian Institutes and ORNL. The Russians focused on batch adsorption studies and completed a broad database on Humosorb interactions with metals, as well as competitive interactions of multiple metals and other cations. ORNL initially confirmed Russian studies with Humosorb binding. The main focus of ORNL work focused on interactions of Humosorb with soil constituents using batch adsorption methods. ORNL also conducted laboratory-scale column studies to test the immobilizing effect of Humosorb under a range of environmental scenarios to determine if combinations of conditions exist that could lead to failure in a field application.
TASK 2. *Greenhouse Studies on Metal Immobilization and Plant Uptake of Metals* -This task was intended to develop a mass balance to determine the extent of leaching of metals from Humosorb- treated soil, and also monitor the uptake of metals by plants. The objectives were to test and confirm

small-scale laboratory studies on Humosorb interactions with metals and soil through longer-term studies on the fate of metals, and to test the effect of Humosorb on plant uptake. This work was to be conducted primarily by the US Industry and the Russian Institute. The focus of greenhouse work by the Russian Institutes was confirmation that their laboratory data on metal complexation and competing cations (Task I) predicts metal immobilization and plant uptake in larger-scale experiments over longer times. Greenhouse studies were also to be conducted by the Industrial partners, with a focus on testing specific hypotheses derived from laboratory studies, particularly those at ORNL on the effects of soil types, soil solution conditions, and coupled processes. ORNL' s role was to assist the Russian and Industry greenhouse studies by developing recommendations on the experimental design, hypotheses to be tested, and interpretation of the data from the greenhouse studies.

TASK 3. *Field Application of Humosorb at Metal-Contaminated Sites* -Humosorb was to be applied to contaminated field sites, and leaching of metals and uptake into plants monitored over long periods of time. The objective of this task was to test predictions of metal immobilization resulting from Humosorb treatment under natural field conditions. It was anticipated that field studies would be conducted by US Industry at contaminated sites in the US, and by the Russian Institute at a contaminated site in the municipal landfill in Moscow. Field studies were not conducted by US Industry. The Russian site was instrumented with lysimeters and subsurface pans to collect soil water for monitoring of metals leaching from the upper soil horizons. Crops were planted and sampled throughout their growth. ORNL assisted in design of the field experiments, especially with respect to site characterization, and the design and installation of monitoring systems for soil- and groundwater.

Problems were encountered in fully accomplishing the goals and objectives of the CRADA. CWE had indicated that they had an agreement with several other US companies who were to actually perform the work specified in the CRADA. Apparently, some problems arose between CWE and the other companies concerning the terms of their agreement. Regardless of the specifics, the effect to the CRADA was that CWE failed to perform any of the work they committed to in the CRADA, and the CRADA was unilaterally terminated by ORNL for reasons of nonperformance in March 1997. Because of the promising results of the work being conducted by the NIS scientists and ORNL, DOE continued funding ORNL' s subcontract with SICPP through it's full two year performance period, which ended in December 1997. The results of the work by ORNL and SICPP will be summarized in this report. CWE provided no data relevant to the deliverables committed to by them in the CRADA either during the CRADA or after it was terminated.

Through actions of the ORNL PI on this CRADA, another US company, Stable Earth Technologies (SET) learned about and became interesting in commercializing Humosorb. This contact was initiated after the CRADA with CWE had been terminated. SET has entered into a joint venture with the scientists at SICPP to manufacture Humosorb in the US and are planning to use the product in remediating 70,000 tons of mercury-contaminated soil. Thus, although some of the technical work specified in the CRADA was not completed due to nonperformance by CWE, the broader objectives of the CRADA-commercialization of a novel remediation technology and diversion of NIS weapons scientists-were achieved.

<u>Benefits to the Fundin2 of DOE Office's Mission</u>

The main goal the DOE/IPP program is development commercially viable technology that will provide employment for NIS weapons specialists and diversion of equipment and facilities from weapons-related production. Therefore this project was specifically aimed to engage scientists and engineers who were directly involved in manufacturing, design and supporting technologies associated with weapons of mass destruction at SICPP. This project employed 16 former weapon specialists from the above mentioned organizations in each of the two years of the project. These scientists and engineers have special knowledge of information regarding manufacture and design of weapons of mass destruction. In the course of this proposed effort we succeeded in redeploying these NIS weapons specialists to the civilian R&D environment, and lay the groundwork for their continued employment in this civilian environment. The last goal will be achieved by utilization of the results of this project to establish long term contacts for these specialists with US and NIS companies working on civilian projects. This project utilized manufacturing equipment and analytical facilities originally built for various defense-oriented projects.

The NIS organization had all necessary infrastructures and capabilities to perform the research and technology development, and they are currently involved in the Defense Conversion Program. This project succeeded in beginning to commercialize new materials and technologies in the US and Russia. We are also planning to develop cooperation between US and NIS industries which will work together to new technologies for environmental remediation.

The project allowed ORNL participation in unique R&D and technologies which became available through the Russian Defense Conversion Program. The joint efforts of ORNL scientists with those in NIS greatly expand the capabilities at ORNL for research in promising new areas in environmental remediation and bioremediation and contaminant fate and transport.

The development of commercial applications is the major priority of the project. In spite of the nonperformance by CWE, another US company was formed after this CRADA was terminated, and this company, Stable Earth Technologies, has formed a joint venture with the former weapons scientists from SICPP. They are beginning to apply Humosorb (which they have re-named "Stabilite") in remediation of mercury-contaminated soil. Further commercialization activities are being pursued that will likely lead to new projects and areas of research in this direction. In particular, the use of Humosorb/Stabilite to remediate soil contaminated with polychlorinated biphenyls (PCB) will be pursued, possibly within the context of another IPP CRADA. PCBs are a very common and wide-spread environmental problem, and one without any currently available technology for cost-effective in-situ remediation, indicated great commercial opportunities for continued employment of former NIS weapons scientists in these environmental remediation activities.

In addition to commercialization within the US, the NIS weapons scientists have been able to achieve commercial success in Russia. The joint stock company formed in Russia by these scientists, Special Biological and Physical Technologies (SBPT), has received $10 million funding in Russia to build a manufacturing facility to produce 10,000 tons of Humosorb per year, and contracts for the sale of this product to the Moscow municipal landfill for preventing leaching of toxic waste. Financial support from this CRADA, as well as technical results developed by SICPP and ORNL during the course of this CRADA contributed to the commercial success of these former weapons scientists.

Technical Discussion of Work Performed

The results of the research conducted by ORNL and SICPP will be briefly summarized below

Characterization of Humosorb: One of the objectives of the project was to extend the chemical characterization of Humosorb already performed by the Russians, and to confirm that the Russian analyses and those performed at ORNL were in agreement. The elemental composition of Humosorb was very similar in analyses by the two groups. The Humosorb received as the raw product contained 48.6% carbon, 30.9% oxygen, 2.9% hydrogen, 1.1% nitrogen, and 66.7% ash. This is very similar to that expected for humic material. Purification by ion exchange reduced the ash content, but did not entirely remove the metals naturally associated with the organic matter. More sophisticated analyses by both groups using 13C- nuclear magnetic resonance and fourier transform infrared spectroscopy confirmed an abundance of hydroxyl and phenolic groups. These functional groups are key to the metal complexation capacity of the humic material. The results of the chemical characterization are consistent with the metal binding capacity of the Humosorb and provides a mechanistic basis for understanding its mode of action.

Binding of Metals to Humosorb: The extent of binding of a variety of metal contaminants was measured by both ORNL and SICPP by means of equilibrium isotherms. Figure 1 shows the extent of binding of mercury, chromium, cadmium and lead four metals. In addition, adsorption isotherms were measured for copper, zinc, nickel and iron. The results confirm and extend previous studies by SICPP concerning the affinity and capacity of Humosorb to strongly complex metals. Studies were also conducted under a range of pH conditions. Metal complexation decreased with decreasing pH, presumably due to competition by protons for cation binding sites.
The extent of binding, the differences in binding affinity between different metals, and the effects of pH on binding are all consistent with the literature on metal complexation by humic substances. The studies conducted in the CRADA represent a significant improvement over the previous Russian data. That data was more anecdotal reports of the amount of metal bound under a single specific concentration or condition. In contrast, systematic studies of metal binding over a range of concentrations (Fig. I) and under different pH conditions (data not shown) provide the scientifically acceptable underpinning required for acceptance of this technology by the scientific community and by regulators.

Competitive Interactions of Metals for Humosorb Binding Sites: In an environmental remediation scenario, multiple cations are present and may compete with each other for binding sites on the Humosorb, possibly reducing the adsorption capacity for the toxic metal of interest. Thus, isotherm experiments were conducted in which three competing metals were present in addition to the cation under study. The non-target ions were introduced at a concentration calculated to bind a total of one third of the total cation binding capacity of the Humosorb (calculated based on the isotherm studies with individual metals described above). At these levels, the presence of competing cations did not affect the adsorption isotherms. These experiments were conducted for all eight of the metals identified earlier. The presence of very high concentrations of major cations, such as calcium can, however, affect sorption of some toxic metals. Although calcium has a relatively low affinity for binding to Humosorb, it can be present in some ground waters at

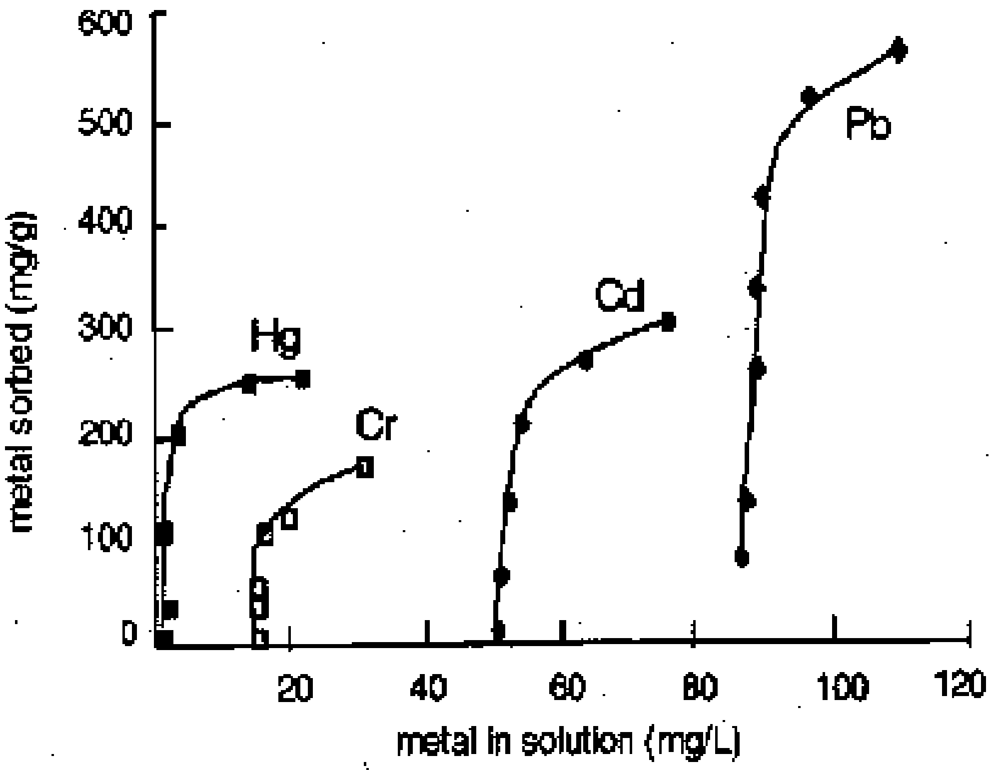


Fig. 1. Adsorption isotherms for metal binding to Humosorb.

concentrations that are orders of magnitude greater than levels of concern for toxic metals. For non- specifically adsorbed metals, such as cadmium, the presence of high levels of calcium reduces binding of cadmium, and addition of calcium can cause release of previously bound cadmium. This is much less of a problem for specifically-adsorbed metals. It is important to recognize this effect because it suggests conditions that could lead to failure of a remediation. For example, it would be inadvisable to lime a cadmium contaminated site treated with Humosorb, even though the resultant increase in pH would be expected to enhance sorption.

Effects of Humosorb on Metal Toxicity and Bioaccumulation by Plants: Greenhouse experiments were conducted to determine the effect of Humosorb on the accumulation of metals by plants and on toxic effects of metals on plant growth. An artificial soil, perlite, was used to avoid complications due to metal interactions with mineral phases of a natural soil. Complex sorption and precipitation reactions could obscure the effects of Humosorb on metal uptake and toxicity in plants. Perlite was loaded into lysimeters with a nutrient solution *(control).* In some lysimeters, toxic metals ranging in concentration from 25 to 250 mg/L were added *(metal-treated).* A parallel set of lysimeters contained the same levels of metals, but Humosorb was also added at a concentration of 1.5% of the volume of the soil *(Humosorb+metal).* The lysimeters were planted with wheat as a model monocotyledon or soybeans as a model dicotyledon. The time-course of plant growth and metal uptake were measured at multiple levels of contaminant. Representative results are presented below.

The presence of the heavy metals resulted in obvious impairment of plant growth (Fig. 2), while treatments with Humosorb+metal exhibited an appearance similar to that of the metal-free controls (Fig. 2). The above-ground portion of the plants were harvested, weighed and analyzed for metal concentration. The metal-treated plants accumulated metals at levels far in excess of that found in the untreated controls (Fig.3). However, the metals levels in the [Humosorb+metal]-treated plants were approximately the same as in the controls. Thus, the Humosorb treatment resulted in >90% reduction in the accumulation of metals in exposed plants (Fig. 3).

The reduction in metal accumulation resulting from the Humosorb treatment was associated with decreased toxicity, measured as the effects of metals on plant growth (Fig. 4). Plant growth was significantly depressed in plants exposed to the metals, but the growth of the [Humosorb+metal] plants was very similar to that of the controls.

Figure 2. Humosorb prevents metal toxicity. Soybeans exposed to Cu, Ni, Zn and Fe (25 mg/L each) show reduced growth and yellowing (left). Soybeans treated with Humosorb and metals (middle) exhibited growth and appearance similar to controls (right).

The competitive interactions of multiple metals on Humosorb' s performance in plant uptake and toxicity were examined. In the sorption studies (above), it was determined that multiple metals did not reduce binding of the target metal as long as the total binding capacity of the Humosorb was not exceeded. The experiments with plants were designed in the same manner as the isotherm sorption studies, but the endpoints were metal accumulation and toxicity (growth in above-ground biomass). Results confirmed that the Humosorb reduces accumulation of metals and protects the plant from growth inhibition when multiple metals are present.

Field Studies of Effects of Humosorb on Metal Mobility and Plant Accumulation and Toxicity: The objective of the field experiments was to demonstrate its ability of Humosorb treatment to bind heavy metals into immobile complexes and prevent uptake into plants under natural conditions. The site of the field experiment was a plot adjacent to the Moscow municipal landfill (Fig 5a). The soil was contaminated with heavy metals, albeit at only moderate concentrations (2.3 mg/kg Cd, 23 mg/kg Ni, 20 mg/kg Pb, 112 mg/kg Cu, 84 mg/kg Cr, and 458 mg/kg Zn). In addition to examining the effect of Humosorb on remediation of the existing metal contamination, additional heavy metals were added to the soil to examine the effectiveness of Humosorb at higher metal loadings. The site was partitioned into 16 treatment blocks that included replicate blocks for controls (no amendments), addition of heavy metals (copper, cadmium, and zinc to total concentrations of 19 g/m1, addition of Humosorb at rates of 5 and 15 kg/m2 (0.5% and 1.5% dry weight, respectively), and addition of both metals and Humosorb. Lysimeters were placed beneath the soil within each plot to collect water leaching through the soil profile for metals analysis. The plots were seeded with clover and grass.
The view of the field site and photographs of representative blocks are shown in Fig. 5. It is visually apparent that plants in the metal-treated

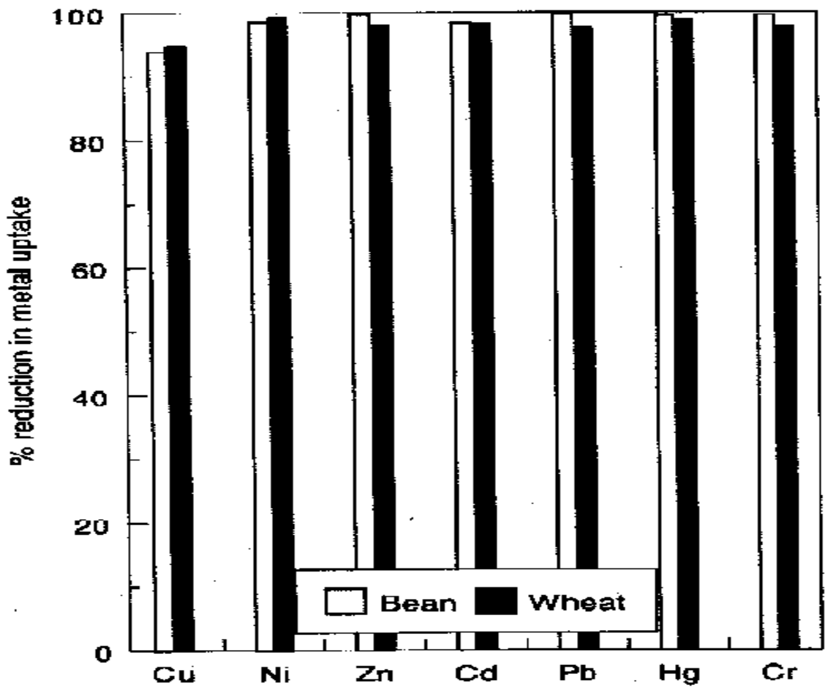


Fig. 3. Metal uptake by Humosorb-treated plants is >90% lower than in treatments without Humosorb (150 mg/L of metals, 1.5% Humosorb)

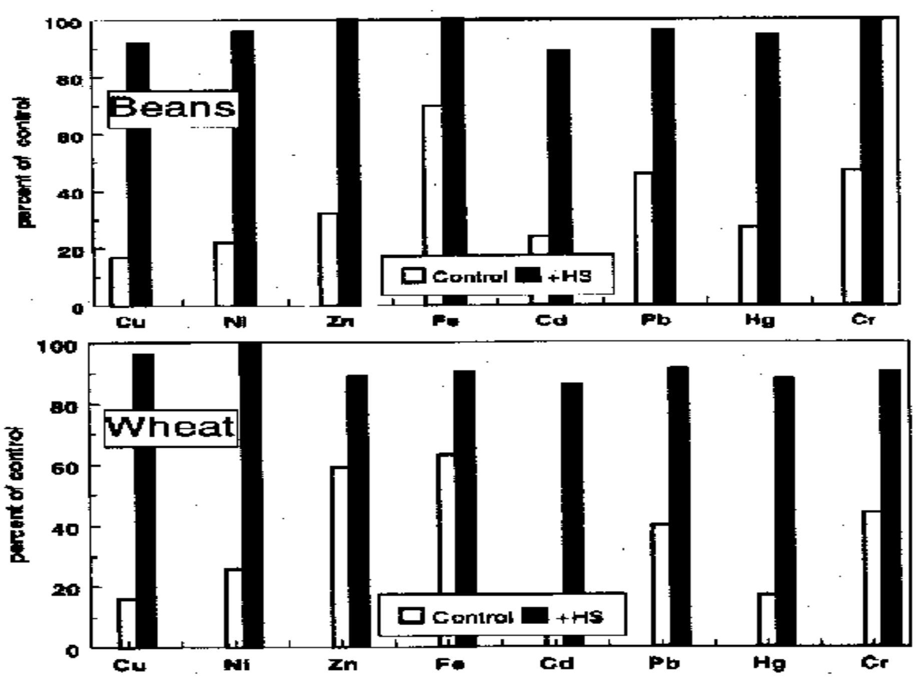
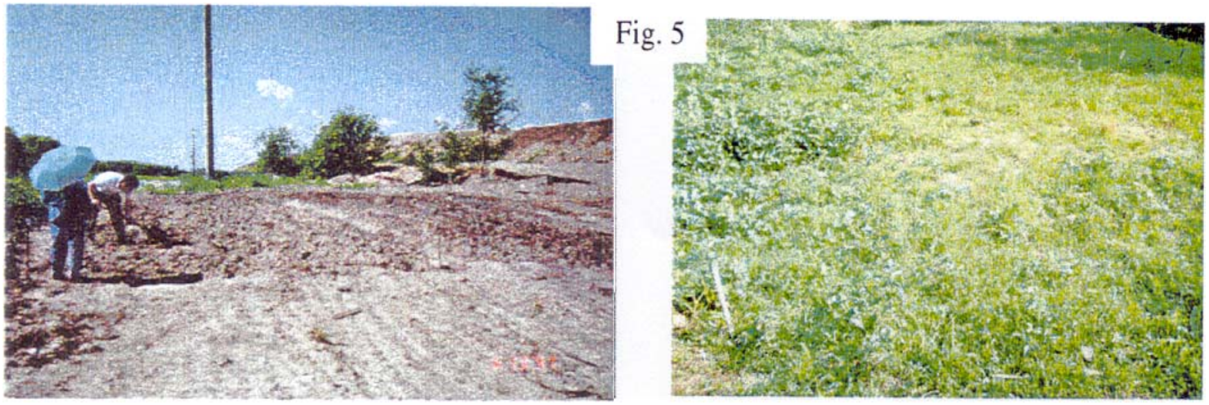


Fig. 4. Humosorb effects on plant growth in presence of metals (150 mg/L). In the presence of 1.5% Humosorb, plant growth is similar to metal-free controls.

Fig. 5



a) View of field site. Landfill lifts are shown in the background. b) A control bed.

c) A bed treated with heavy metals without Humosorb. d) A bed treated with heavy metals and 15 km/m2 (1.5%) of Humosorb.

plot are showing toxic effects which are relieved by treatment with 1.5% Humosorb. Plant growth in the Humosorb-treated plot with the added heavy metals (Fig. 5d) appears more robust than the control plot, which did have moderate levels of metals (Fig. 5b).

The blocks treated with Humosorb leached considerably less metal than either control plots or metal-amended plots (Fig. 6). Levels of metals in water collected in lysimeters beneath the Humosorb-treated control plots were up to 70% lower in metal content than in untreated control plots (Fig. 6a). The effect of Humosorb was only slightly less in the plots that had been amended with 19 mg/m2 of heavy metals (Fig. 6b).

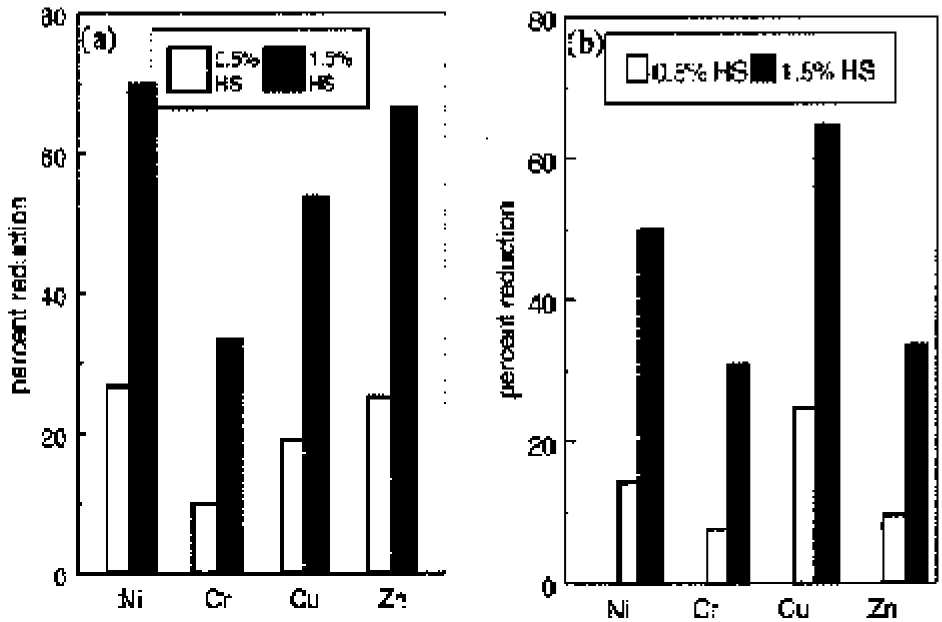


Fig. 6. Percent reduction in the concentration of heavy metals leaching into soil lysimeters in (a) control plots and (b) plots ameded with 19 g/m2 of metals.

Treatment with lower levels of Humosorb (0.5% versus 1.5%) resulted in an approximately proportional less reduction in metal retention by the soil.

Metal uptake in plants (data not shown) and metal toxicity (measured as reduction in plant growth; Fig. 7) were also reduced by treatment of the soil with Humosorb. Treatment with Humosorb had only a slight effect in enhancing growth of plants in the control soil. Metal levels in the soil were apparently not high enough to cause growth reduction (Fig. 7). However, the plots amended with the heavy metals (without Humosorb) exhibited markedly lower growth than the control plants. Addition of the lower concentration of Humosorb had little effect, but treatment with 1.5% Humosorb alleviated the metal toxicity. Plants in the metal-amended soil treated with the higher level of Humosorb exhibited higher biomass in August than the in the control plots. By October, growth in metal-amended, Humosorb-treated plots was not different than in the control plots and only slightly lower than the Humosorb-treated controls (Fig. 7).

The ameliorating affect of Humosorb on plant toxicity is reflected in the reduced uptake of heavy metals by both clover and grass (Fig. 8). Even though Humosorb treatments did not result in large increases in plant growth in the Humosorb-treated controls (Fig. 7), they did result in significant reductions in metal bioaccumulation (Fig. 8a). Although biomass was not affected by the moderate levels of metals in the control soil, the less robust appearance of the plants in the control plots (Fig. 5) may be the result of the accumulated metals, and the healthier appearance of the Humosorb-treated plants (Fig. 5d) may reflect the reduction in plant uptake due to Humosorb treatment.

The fractional reduction in metal bioaccumulation in the metal-amended plots (Fig. 8b) is similar to, and for many metals, much greater than that in control plots (Fig. 8a). Higher levels of Humosorb resulting in proportionally greater reductions in bioaccumulation. It appears that the reduction in metal uptake effected by the 0.5% Humosorb treatment was not sufficient to relieve the toxic effects of the metals on plant growth (Fig. 7).

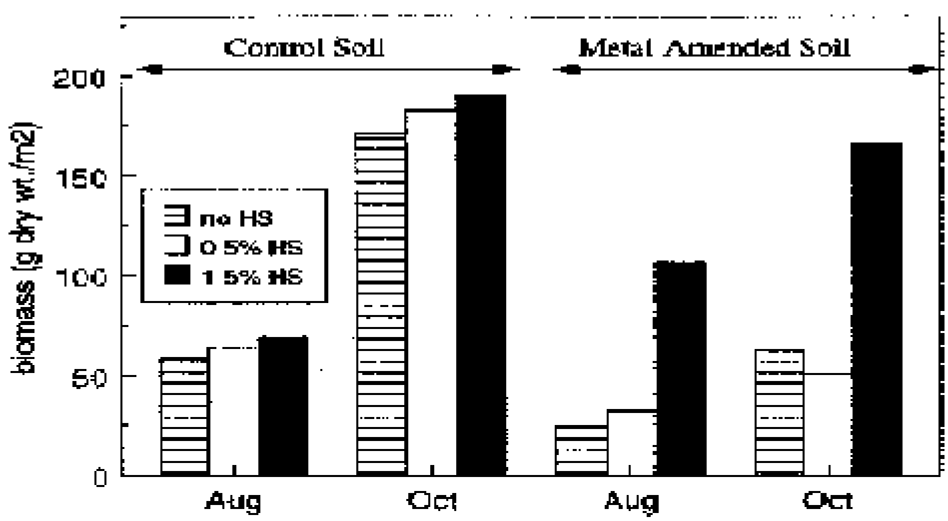


Fig. 7. Effects of Humosorb treatment on plant growth in control and metal-amended plots.

Summary of Technical Results: Humosorb was extensively characterized by a variety of chemical methodologies. Binding of 8 heavy metals was determined by means of batch isotherms, and competitive effects of multiple metals were

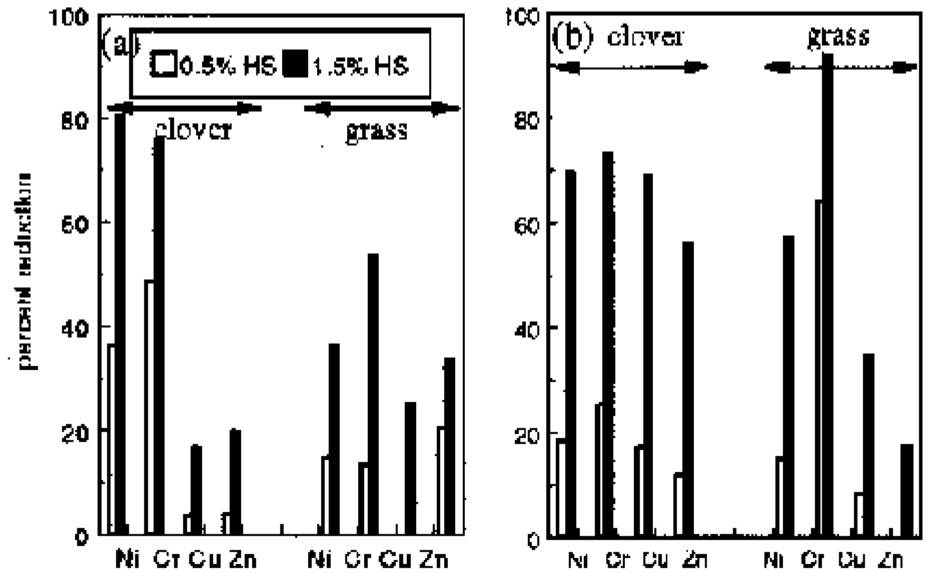


Fig. 8. Humosorb treatment results in reduction of metal uptake in clover and grass in (a) control plots, and (b) metal-amended plots.

examined. These data provide the scientific basis for understanding the mode of action of the technology. Greenhouse experiments demonstrated that Humosorb reduced the accumulation of metals by plants and relieved the toxic effects of the metals on plant growth. A dose-dependent response between increasing Humosorb levels and the magnitude of ameliorating effects on accumulation and growth provide additional evidence that the causative agent was the Humosorb treatments. A field study was conducted at the Moscow municipal landfill to determine if the effects of Humosorb on metal mobility, bioaccumulation and toxicity observed in the laboratory setting were apparent over large temporal and spatial scales of natural field situations. Field results confirmed that metal mobility was significantly lower in the Humosorb-treated plots, and that plant bioaccumulation and toxicity were reduced. The laboratory, greenhouse and field studies conducted within this CRADA provide convincing evidence that Humosorb can stabilize metal-contaminated soil and remediate plant toxicity and bioaccumulation. Besides the benefits of reduced metal uptake to the health of the plants themselves, there are implications to human health. For example, these results provide strong evidence that pasture contaminated with radionuclides from Chernobyl could be returned to productive and healthy dairy production by treatment with Humosorb. Radionuclides would be sequestered in the soil rather than being transferred up the food chain from contaminated grass to cows and then to milk consumed by humans.

Inventions (Made or Reported)

No inventions were made or reported.

Commercialization Possibilities

Commercialization possibilities are excellent and are being implemented

A US company, Stable Earth Technologies (SET) was formed after this CRADA was terminated, and this company has entered into a joint venture with the former weapons scientists from SICPP. They are beginning to apply Humosorb (which they have re-named "Stabilite") in remediation of 70,000 tons of mercury-contaminated soil. SET will be manufacturing Stabilite in the US to produce the product needed for this remediation. They have verbal agreement for two other remediation contracts, and are actively seeking additional contracts for commercial applications of the technology.

In addition to commercialization within the US, the NIS weapons scientists have been able to achieve commercial success in Russia. The joint stock company formed in Russia by these scientists, Special Biological and Physical Technologies (SBPT), has recently received $10 million funding in Russia to build a manufacturing facility to produce 10,000 tons of Humosorb per year, and contracts for the sale of this product to the Moscow municipal landfill for preventing leaching of toxic waste. Financial support from this CRADA, as well as technical results developed by SICPP and ORNL during the course of this CRADA, contributed to the commercial success of these former weapons scientists.

Thus, even though not all the work specified in the CRADA was completed due to nonperformance by CWE, the broader objectives of the CRADA-commercialization of a novel remediation technology and diversion of NIS weapons scientists-were achieved. Addition technical work would be required to provide the scientific validation and testing required to achieve the full commercial potential of Humosorb (Stabilite) for remediation of metal-contaminated soil. SET is interested in pursuing a Thrust II IPP project with ORNL and SICPP to complete the full technical scope of work specified in the original CRADA.

Plans for Future Collaboration

The Russian weapons scientists have formalized an ongoing commercial business agreement with the US company, SET. In addition, SET, SBPT , SICPP, and ORNL are pursuing a follow-on Thrust II IPP project to complete work not executed during the original CRADA due to the nonperformance problems with CWE. They are also proposing a new application of Humosorb {Stabilite) for in-situ remediation of PCB contaminated soil. PCBs are a very common and wide-spread environmental problem, and one without any currently available technology for cost-effective in-situ remediation, indicated great commercial opportunities for continued employment of former NIS weapons scientists in these environmental remediation activities.

Conclusions

This CRADA arose through the United States Industrial Coalition, Inc. to foster new business partnerships between the US and the Newly Independent States of the Former Soviet Union under DOE's Incentives for Proliferation Prevention (IPP) Program. The main goal the DOE/IPP program is development commercially viable technology that will provide employment for NIS weapons specialists and diversion of equipment and facilities from weapons-related production. The NIS scientists involved in this CRADA had developed-and sought assistance in commercializing-a process for efficient and cost-effective extraction of humic acids from brown coal. The resulting product was called "Humosorb". The goal of this project was to evaluate and field validate the application of Humosorb for in-situ remediation of meta1- contaminated soils, specifically by immobilizing heavy metals in contaminated soil and prevent their uptake into plants.

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Significant technical progress was made by ORNL and the Russian scientists in testing and validating the use of Humosorb in metal remediation. The product was fully characterized with respect to its chemical and physical properties. The extent of immobilization of eight heavy metals to Humosorb was determined and the effect of complex mixtures of toxic metals was also evaluated. Greenhouse studies demonstrated that Humosorb causes very large reductions in the bioaccumulation of metals by plants and a corresponding decrease in plant toxicity. In a field demonstration experiment, the mobility of heavy metals in soil was decreased by application of Humosorb and the protective effects to plants seen in the greenhouse studies were also observed at the field scale.

Work intended to be performed by the US Industrial partner, greenhouse studies on metal mobility and field studies at contaminated sites in the US, was not performed. The greenhouse studies were to have provided a mass balance demonstrating the extent to which Humosorb reduced metal mobility in soil, with a focus on the effects of soil types, soil solution conditions, and coupled environmental processes. The loss of additional field experiments is even more problematic. The original scope of work sought to increase confidence in the performance of Humosorb by conducting field studies at multiple Russian and US sites that offered contrasts in soil conditions or the nature and extent of contamination. Without these additional studies, the initial commercial applications of Humosorb (Stabilite) must be viewed more as feasibility demonstrations rather than application of a mature technology. Commercialization is likely to be slowed by this short-coming.

Nevertheless, the principal objectives of this DOE/IPP Program have been achieved as a result of this CRADA. The main goal the DOE/IPP program is development commercially viable technology that will provide employment for NIS weapons specialists and diversion of equipment and facilities from weapons- related production. In spite of the nonperformance by the original CRADA participant, CWE, another US company was formed to commercialize Humosorb for environmental remediation in the US. This company, Stable Earth Technologies, has formed a joint venture with the former weapons scientists from SICPP, and are beginning to apply Humosorb (which they have re-named "Stabilite") in remediation of mercury-contaminated soil. Further commercialization activities are being pursued that will likely lead to new projects and areas of research in this direction. In particular, the use of Humosorb/Stabilite to remediate soil contaminated with PCB will be pursued, possibly within the context of another IPP CRADA. In addition to commercialization within the US, the NIS weapons scientists have been able to achieve commercial success in Russia. Financial support from this CRADA, as well as technical results developed by SICPP and ORNL during the course of this CRADA contributed to the commercial success of these former weapons scientists.

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