USGS

WATER RESOURCES RESEARCH GRANT PROPOSAL

Title: Development of an Innovative Kenaf-Based Water Treatment Process

Focus Categories: GW, TRT, TS

Keywords: Hazardous wastes, groundwater quality, pollution control

Timeline: March 1999 - February 2001 (Two Year Duration)

Federal Funds Requested: $5,090 (YR1) and $5,097 (YR2)

Nonfederal funds committed: $ $15,910 (YR1) and $15,903 (YR2)

Congressional District: No. 3 - Mississippi

Principal Incestigator: Dr. Mark E. Zappi, P.E., Director of the Environmental Technology Research and Applications (E-TECH) Laboratory, Dave C. Swalm School of Chemical Engineering, Mississippi State University (MSU)

Research Need

Many of the contaminated surface waters and groundwaters currently undergoing treatment or scheduled for treatment contain organic contaminants at relatively low levels (sub-100 ppm levels); yet, these levels are still well above regulatory requirements requiring treatment prior to discharge. Recent pollution prevention initiatives within industry have resulted in wastewaters that do contain lower levels of contamination; however, these waters still contain contamination levels requiring treatment. The treatment of these waters pose both technical and economic challenges to industry and site owners (Acar and Zappi 1995). With regard to Mississippi, many industries are small businesses that are already stressed by the challenges of today’s market conditions. Having to face complex environmental compliance issues, such as water treatment, only further exasperates their situation. However, these water sources do require treatment to preserve/restore the resource quality (and keep the company in compliance).

Currently available treatment technologies usually have problems treating these waters. Typically, these influents do not contain sufficient organic substrates to adequately support biotreatment units (Zappi et al. 1993). Many of these chemicals cannot be air stripped because of low volatility (Haas and Vamos 1995). Plus, current regulatory guidance requires treatment of air exiting air stripper units due to concerns over air pollution. Chemical oxidation offers an alternative; however, these techniques are developing, somewhat costly, and often require highly trained operators due to system complexity (Langlais et al. 1991). Also, the presence of hydroxyl radical scavengers within the influents and poor influent UV transmissivity adversely impacts oxidation process performance (Zappi 1995). Activated carbon adsorption offers a viable option.

However, activated carbon has limitations in that spent carbon must be transported off-site for disposal or regeneration. Therefore, the state of affairs facing engineers and scientists attempting to treat low level contaminated waters using the above discussed technologies results in the selection of a process that has significant short-comings. A technically superior, yet operationally simpler treatment process within a reduced cost bracket is needed to assist industries in meeting environmental regulations. An operationally-simple system would improve the quality of endangered water sources because of increased usage by industry resulting in cleaner discharges.

Proposed Process

A novel process is proposed that will address the shortcomings of the technologies discussed above, while meeting the needs of industry. The envisioned process involves adsorption of organic contaminants onto crushed kenaf fibers. Kenaf is a plant that is in the same plant family as okra. Kenaf is an agricultural crop that has recently been found to have many uses including animal litter, paper manufacturing, and composite material construction. Recent unfunded research at MSU indicates that kenaf has a high sorption capacity for organic contaminants (discussed later). The fibers of the kenaf plant are resistant to water logging and are structurally stable when exposed to overburden loads allowing tight, yet sturdy packing. Kenaf fibers will be packed into a column where contaminated water will be passed through the column using up-flow hydraulics to provide intimate contact between contaminated water and the kenaf fibers. The contaminants will adsorb onto the sorptive sites on the kenaf and the column operated until all of the kenaf fibers within the column are completely spent (i.e. all sorption sites filled). This approach is very similar to that used with activated carbon adsorbers; however, once the kenaf becomes spent, then the fibers will be removed and placed into a compost bed (see attached drawing). The compost bed will be used to reduce waste kenaf volume and degrade the adsorbed chemicals via biotreatment.

Research Benefit: Benefits expected with the development of the proposed process are:

a. The kenaf-based biosorptive process is expected to be much cheaper than activated carbon in that kenaf is a renewable resource that is easily cultured within the Southeastern United States. MSU is actively involved in the discovery of innovative uses of kenaf. This effort is managed under the MSU Kenaf Research Program housed within the Department of Agriculture and Biological Engineering. Past research efforts by the MSU Kenaf Research Team have refined growing protocols and plant material processing techniques to economically grow and process kenaf. The plant fibers are easily prepared using processing techniques that economically wash, crush, and segregate the various fractions. Alternatively, activated carbon requires aggressive processing of carboneous materials (nutshells, bone, or coal) that involves high temperatures and pressures; both of which are very costly. Cost estimates place carbon at $1.50 versus less than $0.10 per lb. kenaf.

b. Kenaf fibers are easily stored and extremely stable under high impact and overburden stresses. Activated carbon is easily crushed to useless fines under similar handling conditions. In fact, to retain structural stability, activated carbon is usually loaded into and out of adsorber vessels using water-carbon slurries which increases system complexity and cost.

c. Composting is a well developed process that has been widely used for disposal of plant debris and stabilization of municipal biosolids. The final product from compost systems, including the type proposed within this process, is a humus-based material that has a marketable value as a agricultural amendment (Maynard 1995). Using composting to dispose of the spent kenaf fibers will also result in the on-site degradation of the adsorbed contaminants meaning that the proposed process is a destruction treatment process that will eliminate the pollutants as an environmental and regulatory problem. Carbon must be transported off-site and regenerated using special equipment that utilizes high temperature and pressures to destroy the contaminants.

Objectives

The overall objectives of this project will be to develop and prove the utility of the kenaf biosorption process for treating contaminated waters. Secondary objectives to be addressed include:

a. Evaluation of the adsorptive capacity of candidate kenaf fibers composed of various plant fractions and/or fibers made from differing processing techniques for three phenolic compounds (phenol, dichlorophenol [DCP], and pentachlorophenol [PCP]). The rationale for selecting phenols will be discussed in the next section.

b. Evaluation of the stability of the various fibers within saturated column reactors under long-term, dynamic operational flows.

c. Evaluation of various composting approaches for both reduction of plant mass and degradation of phenolic compounds.

d. Determination of the extent of mineralization achieved for the phenolic compounds within the compost reactors.

e. Evaluate the impact of chlorination level on the rate of adsorbed phenolic compound degradation within the compost reactors.

The major emphasis of the proposed effort is to develop an alternative treatment technology, kenaf-based biosorption, that has technical and economic characteristics that are not provided by current processes. The attractiveness of this effort is that the developed process will have applicability for treatment of adsorbable and biodegradable organic contaminants (many USEPA listed contaminants fall into this category). Therefore, to facilitate process development, a target contaminant must be selected for use as a test pollutant. Phenols were selected as the target contaminants for this study. Phenols were selected for several reasons which include: [a] phenols were listed by the USEPA in the 1993 Toxics Release Inventory (USEPA 1995) as one of the top 25 chemicals most discharged by US industries (This report also states that the Southern US (TX, LA, MS, AL, and TN) was among the largest contributors to these releases), [b] phenols are listed on many target regulatory lists as a contaminant of primary interest, and [c] phenols have been the subject of many research topics in the past; unfortunately, most of this work was targeted toward high level contaminated waters with little attention focused on low level contamination that now constitutes a large fraction of the phenolic mass released into the environment or discharged into POTWs.

Phenols are biodegradable with increasing chlorination posing more difficulty for biotreatment (Baker and Herson 1994). Unfortunately, low level phenolics contaminated waters usually do not contain a sufficient organic loading to properly sustain an active aerobic bioreactor (Zappi et al. 1993). Of the three phenols, PCP is the most refractory to biotreatment (Hurst et al. 1997) due to enzyme inhibition posed by the chlorines.

SUMMARY OF TECHNICAL APPROACH

The research objectives of this project will be met through performance of a series of research tasks that are briefly summarized below:

Analytical Techniques: Note that the phenolic compounds and associated biodegradation by-products will be analyzed during this study using a Model 1100 Hewlett-Packard High Performance Liquid Chromatography (HPLC) unit equipped with a diode array detector following USEPA approved analytical methods. Chloride production, oxygen, and system pH will be monitored using specific ion probe technique. General water constituent analyses will be performed using methods described in the 19th Edition of Standard Methods.

Project QA/QC - A research project is only as good as the quality of the data collected during experimentation. A QA/QC Plan will be drafted generally following techniques recommended by the USEPA for performance of their research efforts. Appropriate standards and instrument calibration will be performed following equipment manufacture recommendations. Also, extensive test controls will be maintained throughout the various experiments to help identify actual removal mechanisms, along with sufficient experimental sample replication.

Task 1 - Evaluation of Adsorption Capacity for Candidate Kenaf Fibers - The ability of kenaf to adsorb the three phenolic compounds will be evaluated using various fibers manufactured from several plant processing techniques already developed by MSU. The sorptive capacity will be determined using traditional adsorption isotherm techniques. Initially, adsorption kinetics will be evaluated by generation of time-based sorption plots which will be used to select appropriate shake times for the isotherm tests. Precleaned 500 ml all-glass bottles will be filled with the candidate kenaf fibers and phenol solutions and agitated using a recipitating table. Isotherms will be generated for each phenol and associate candidate kenaf fiber source. Also, an isotherm will be developed for each phenol using a test solution containing all three phenols to evaluate potential competitive adsorption effects. Fruendlich constants will be calculated using appropriate graphical methods. Each series of tests will be performed using triplicate bottles.

Task 2 - Kenaf Fiber Stability During Dynamic Column Operation - The stability of the various kenaf fibers will be tested by loading the fibers into 2 inch ID by 24 inch long all-glass columns capped with teflon plugs. Each fiber source will be evaluated in duplicate columns. The columns will be operated by flowing tapwater up through the columns to achieve an empty bed contact time of one hour. Stability will be evaluated by measuring weight losses for dried fiber mass dried at 65°C and changes in column headloss as measured via pressure differentials. Additional testing protocols will be investigated for evaluating fiber stability. Tests used for evaluating packing paper stability under wetted conditions will be reviewed that may also prove useful for inclusion in this study.

The impact of extreme pH conditions that may be encountered with some industrial wastewaters will be evaluated using batch shake tests composed of kenaf fibers and aqueous solutions buffered to achieve pHs of 2, 4, 8, and 11. All-glass bottles (500 ml capacity) loaded with 300 ml 30% (w/w) fiber-water slurries will be used in these tests. Weight loss for fiber retained on fiber-specific sieves (the sieve number will be based on the starting nominal diameter of the kenaf particles) will be used to evaluate pH stability. These experiments will be performed using triplicates and agitation provided using orbital shaker tables.

Task 3 - Composting of Spent Kenaf Fibers - The degradation rates of both kenaf fibers and phenolic compounds within aerobic and anoxic bench-scale compost systems will be evaluated. Temperatures within the bench-scale compost reactors (one liter units) will be maintained within mesophilic or thermophilic regions using outer water jackets that will be circulated with water heated to appropriate levels (ramped to approximately 30°C or 55°C over time which simulates/tracks biotic-based compost reactor heating). Moisture levels will be maintained at least 40% which is commonly used with windrow systems and aeration provided using air stones buried in the compost piles. Various compost amendments that are typically used at composting operations for disposal of garden and wood debris will be tested initially for fiber volume reduction (kenaf degradation) using clean fiber samples. Examples include manure, biosolids, vegetable wastes, nutrient broths, municipal compost, and aged humus. Amendment dosing will be evaluated using mass-based percentage inputs. Kenaf material degradation rates will be determined using units of kg kenaf mass per time. Also, total volumetric reduction will be determine based on a solids balance. Compost operations will be optimized during this phase based solely on kenaf mass degradation (i.e. volume reduction). Later, additional optimization may be performed, if necessary, to accelerate both the rate and extent of phenol degradation. This approach will be used because it is hypothesized that it will be more difficult to reduce kenaf mass than to degrade the phenols. However, this hypothesis may be disproved during testing upon which compost operations will be tailored around phenol removal which is most important.

Based on the results of the volume reduction experiments and the tests described in Tasks 1 and 2, an optimal kenaf fiber will be selected. This fiber will be contaminated with each of the three phenols separately to evaluate the potential differences in biodegradation rates based on the level of chlorination. Spent kenaf will be manufactured by batch contacting clean kenaf with appropriately dosed water samples that will be agitated for at least three days. Additional experimentation will be performed to study compost dynamics using kenaf fibers spent with all three phenolic species. Each compost experiment will be performed using duplicate reactors.

Task 4 - Evaluation of Contaminant Mineralization - The extent of phenolic compound mineralization achieved during composting of the spent kenaf will be determined using radio labeled pentachlorophenol (^{14}C) following techniques similar to those reported by Larsson and Lemkemeier (1989). Due to the high cost of labeled compounds, this effort will only evaluate PCP mineralization. PCP was selected because it is widely accepted as being the most difficult phenol to biodegrade. If PCP is successfully mineralized, then the other phenols should also be at least similarly mineralized.

These experiments will be performed in triplicate using 500 ml Bellco brand shakeflasks equipped with side-arm CO_2 traps charged with potassium hydroxide. The compost system will be operated using conditions deemed optimal from the above tasks. Acidification after reaction times are completed will be performed to convert all carboneous inorganic species to CO_2. Also, a final flask acid washing will ensure little or no labeled species are left sorbed onto glass surfaces. Mass balance of labeled compounds will be performed via analysis of both solid-slurries and air phases using a Packard Tri-Carb 2100TR scintillation counter with appropriate Ultima Gold™ cocktail formulations.

Task 5 - Dynamic Column Runs - A series of dynamic column runs will be performed using the optimized process based on the results from the above tasks. This experimentation represents final process verification prior to pilot studies. Duplicate columns will be operated for treatment of DCP and PCP contaminated influent. The columns will be operated until complete breakthrough of both phenols occurs at which the spent kenaf will be composted. The residuals from composting will be analyzed for phenolic content.

Task 6 - Cost Analysis of the Developed Process - An economic evaluation of the various potential design options for the developed technology will be performed. This evaluation will generally follow traditional engineering economics (Newman 1980) which will provide costs associated with capital investment, O&M requirements, and total unit pricing ($/kgal treated).

Task 7 - Draft Final Report - A final report will drafted that details the results of the experiments, proposes a protocol for system design, and presents an evaluation of relative process economics. A first draft will be prepared for review and appropriate corrections and modifications made prior to submittal of the final report for publication by MSU. Note that an interim report will be drafted for agency review. The expected date for the submittal of the interim and final reports are January 2000 and January 2001, respectively (See Milestones Section below).

PROJECT MILESTONES/SCHEDULE

Two major milestones will be completed within the requested two years. The first will be the completion of the majority of separate mechanism-based research (i.e. Tasks 1 -3). The second year will focus on mineralization studies and performance of the dynamic column studies. A project timeline for the proposed effort is detailed below:

Task	Date Expected to be Completed
1. Evaluation of Adsorption Capacity	Aug/99
2. Evaluation of Kenaf Stability	Oct/99
3. Composting of Spent Kenaf	Jan/00
4. Evaluation of Mineralization	Feb/00
5. Dynamic Column Runs	Dec/00
6. Complete Final Report	Jan/01

Note that an interim report will be drafted and submitted for review in January of 2000. This report will likely be followed by the publication of at least one peer-review paper and presentation of results at two conferences.

RESEARCH FACILITIES

The Environmental Technologies Research and Applications (E-TECH) Laboratory of the Department of Chemical Engineering at Mississippi State University is a well equipped, modern laboratory that has houses a wide variety of research projects on remediation technologies. This laboratory is the focal point of the MSU Department of Chemical Engineering's Graduate Environmental Engineering Program. The facility mission is to support government and industry in their environmental activities through performance of both basic and applied research. The facilities within the E-TECH Laboratory are more than adequate to fully support the proposed research efforts. The E-TECH Laboratory is equipped with GC, GC/MS, HPLC, stopped-flow spectrophotometer, IR spectrophotometer, ISE meters, DO meters, and standard spectrophotometers. The research team will also have access to an IC, BET surface analyzer, liquid scintillation unit, TOC analyzer, E-Microscope, XPS system, and AA units found within other MSU departments. The E-TECH Laboratory also maintains various AOP reactor systems, biocell reactors, bioslurry reactors, ozone generators, fermenters, air phase ozone monitors, and incubators. The Department of Chemical Engineering also houses a fully equipped machine shop and computer laboratory for production of specialty reactors and equipment with full computer interfacing. One experienced engineering technician is employed year-round by the department.

Mississippi State University is also home of the Diagnostic Instrumentation and Analytical Laboratory (DIAL), the Mississippi Technical Assistance Program (MISSTAP), Forest Products Laboratory, and the State of Mississippi's Chemistry Laboratory. These groups can be contacted for collaborative efforts and advice if the need arises.

PAST RELATED RESEARCH EFFORTS

Of most interest to this preproposal in terms of proving that our concept is indeed a viable and realistic process that can be developed are the results of some recent unfunded, exploratory research undertaken by Dr. Zappi within his laboratory. Using kenaf fiber made primarily from inner pulp, Dr. Zappi's group generated an adsorption isotherm for dichlorophenol. The loading derived from this effort was compared to the DCP loading generated by Dobbs and Cohen (1980) for removal of DCP using commercial activated carbon. The data indicated that kenaf has an adsorptive capacity for DCP very similar to that of activated carbon.

Past studies that evaluated the sorptive ability of plant mass have shown this capability to be appreciable (Best et al. 1997). By having a similar sorptive capability as carbon along with cheaper manufacturing, processing, and spent adsorbate disposal techniques (i.e. composting), the potential for successful development of the technically and economically viable process proposed in this preproposal is believed to be extremely high.

The use of novel adsorbents is not new. Kim et al. (1997) used waste tire rubber as an effective adsorbent for removing chlorinated solvents. Eichenmuiller et al. (1997) used porous polymers to remove acenaphthene from water. Srivastava et al. (1997) evaluated using waste carboneous particles from a fertilizer production facility for removing dichlorophenol from aqueous solutions. All of these innovative approaches showed promise; however, in each case, all adsorbents had to be regenerated using either high temperatures (much like activated carbon) or chemical dissolution using aggressive chemicals that did not ultimately result in the destruction of the contaminant. The kenaf-based biosorptive process differs from these processes in that composting, a technology proven to be much cheaper to implement than thermal processes, will be used to destroy the spent adsorbents and sorbed adsorbates.

Composting is a well-established process that is gaining increased attention for its simplicity and effectiveness (Goldstein and Steuteville 1996). Composting of the spent kenaf is expected to be successful for both the reduction of spent kenaf fiber volume and the degradation of the adsorbed phenols. Several well-documented studies clearly showed that compost systems are very capable of rapidly achieving high levels of volume reduction of plant wastes many of which were more recalcitrant than kenaf due to their composition of more stable cellulose and high bark content (Reinhart and Tanner 1995). Liao et al. (1995) reported volume reductions after composting in excess of 50% and 40% for sawdust and peat moss, respectively.

In terms of organic chemical degradation, composting has been successfully used for degradation of very complex organic compounds including explosives (Tuomi et al. 1997), herbicides (Dooley et al. 1995), biosolids (Reinhart and Tanner 1995), and petroleum wastes (Epstein and Wu 1997). In fact, Semple and Fermor (1995) determined that composting was effective in the treatment of chlorophenol contaminated soils. Finally, since the proposed treatment process must be operated year-round, including winter months, some concern was felt toward the ability of a compost system to properly function during this period. However, Lynch et al. (1997) proved that composting during severe winter conditions is not only possible, but successful.

In summary, a novel adsorbent is proposed for treating low-level contaminated water sources. To convert this physical water treatment process to an on-site destruction technology, a well-established biodegradation mechanism (composting) will be used. Both laboratory studies recently performed by this research team on DCP/kenaf adsorption and the reviewed literature indicates that our proposed concept is scientifically well based and has potential for dramatic cost savings.

PI RESEARCH BACKGROUND

A summarized CV for Dr. Zappi is provided as an addendum to this preproposal. However, a brief description of Dr. Zappi's background is provided below:

Mark E. Zappi, Ph.D., P.E. - Dr. Mark Zappi, an Environmental Engineer with the faculty of the Department of Chemical Engineering, will serve as project PI. Dr. Zappi has extensive experience with developing innovative processes for waste treatment. During his tenure at his former position at the USAE Waterways Experiment Station, Vicksburg, MS, Dr. Zappi was head of the Biological and Oxidation Processes Team. Dr. Zappi has performed development and optimization research on several treatment processes including bioslurry, composting, solid phase oxidation (SlurOx), oxidation preconditioning prior to biotreatment, UV oxidation, carbon adsorption, phytoremediation, ion resin adsorption, and biofiltration. Dr. Zappi has worked on treatment process-related research ranging from evaluation of basic reactions that control the effectiveness of the process to the design and testing of innovative pilot-scale systems in the field. Dr. Zappi has been involved in over 80 contaminated site remediation projects. This "real-world" experience will be used to further the application potential of the candidate process under development in this project. Dr. Zappi is also an Associated

Editor for the Journal of Environmental Engineer and is a peer-reviewer for numerous other environmental technology and scientific journals. This experience with both conference and journal publishing has allowed Dr. Zappi to have an excellent pulse on the technology and will prevent redundant research efforts during the implementation of the proposed research effort.

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