Novel Materials for Facile Separation of Petroleum Products from Aqueous Mixtures Via Magnetic Filtration

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Title: Novel Materials for Facile Separation of Petroleum Products from Aqueous Mixtures Via Magnetic Filtration
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Research Category: Separations

Description:

The major objective of this research is to develop magnetic extractants that can be used in conjunction with a magnetic filter to efficiently and economically remove petroleum products from aqueous solutions and mixtures. The project will synthesize three types of magnetically-active materials that are capable of absorption of hydrocarbons so that the latter can be rapidly and efficiently removed from water using magnetic filtration technology. Initial tests will be performed with mixtures of hexadecane, a typical alkane, and phenanthrene, a typical polynuclear hydrocarbon. These compound will allow testing of the performance of the magnetic extraction process on pure compounds in a manner that is conducive to a better theoretical assessment of the process. At the same time, a more practical assessment will be made using crude oil/water and crude oil/brine mixtures. The primary investigator (P.I.) will also obtain at least two actual contaminated water samples from the petroleum industry (e.g. drill cuttings, produced water, etc.). Oklahoma State University has a close research interaction with Halliburton that will provide a vector for obtaining these samples. The ability to purify such "real-life" samples in the laboratory will provide the necessary proof of the applicability of magnetic filtration to the petroleum industry. A second goal of the research is to test whether the magnetic extractants in conjunction with an applied magnetic field would be useful for breaking emulsions.

This proof of concept research will demonstrate the feasibility of a magnetic extraction process and determine the parameters that will allow an assessment of the potential economic benefit to the petroleum industry. Ultimately, achievement of these goals is expected to benefit the petroleum industry through a reduction in the cost of compliance with U.S. environmental regulations. Furthermore, the research will involve both a graduate student and an undergraduate student and will thus contribute to the human resource needs of the petroleum industry in the very important area of environmental compliance.

The deliverables of the proposed research are the following. First, three types of potential magnetic extractants, each with two different types of binding groups (a 18-carbon alkane and biphenyl) will be synthesized. "Magnetic" adsorption isotherms for two test petroleum products, an alkane and a PAH will be measured. The three magnetic extractants that exhibit the best extraction behavior will be tested for removal of crude oil from water and brine by magnetic filtration. The two most successful extractants at this stage will be tested in the separation of hydrocarbons from at least two actual industrial samples and for the breaking of an oil in water emulsion. The regeneration and reuse of the magnetic extractants using a magnetically-assisted washing step with petroleum ether will also be demonstrated using crude oil-loaded extractants.

Progress Summary/Accomplishments:

The main task for this period was completing the preparation of magnetic extractants and testing them for removal of organic compounds from water. We developed a method for making magnetically-active activated carbons. These materials are simply prepared by impregnating paper towels with an aqueous solution of iron gluconate or an iron gluconate/nickel gluconate mixture and then firing at 500˚C under a nitrogen atmosphere. The materials thus produced have surface areas in the range of 40-50 m^2/g. Nanocrystalline magnetite and nickel ferrite are detectable by X-ray diffraction in the solids and, as expected, the ferrite-containing material exhibits the strongest response to a magnetic field. Several extractants based on polydimethylsiloxane/iron or iron oxide composites were also prepared since a recent report indicated that polydimethylsiloxane is a good absorbant for phenanthrene.

The testing of the extractants was initiated using a 104 ppm aqueous solution of decane. 20 g of solution was treated with 0.5 g of extractant. After filtration, 6 g the aqueous solution was extracted with 1 g hexane and the extracts were analyzed for decane by GC/MS. The results are as follows:

Extractant	Final Decane Concentration	Extent of Decane Removal
Magnetite/Activated Carbon	61.2 ppb	99.94%

Nickel Ferrite/Activated Carbon	59.1 ppb	99.94%
Polyoctadecylmethacrylate/iron	93.9 ppb	99.91%

Thus, the three extractants tested thus far have performed very well and can reduce the concentration of decane, a representative alkane, to the parts per billion level. It is expected that less water-soluble alkanes will partition even better.

Future Activities:

Future activities will include preparation and characterization of higher surface area magnetically-material/activated carbon composites by use of a cellulosic material other than paper.. Testing of the magnetic extractants for decane and phenanthrene removal from water will be completed.