



Use of GreenZyme® for remediation of porous media polluted with jet fuel JP-5

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ABSTRACT

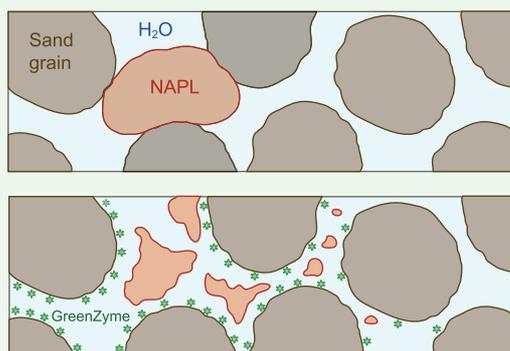
Jet fuel may be released in the environment either by in-flight fuel jettisoning (fuel dumping) or accidentally from spills and leaks, and eventually can reach subsurface formations where it can remain as long-term source of pollution. Remediation of aquifers contaminated by jet fuels is not a trivial task. This experimental study examined the effectiveness of a water-soluble, DNA-protein-based biodegradable non-living catalyst, with commercial name GreenZyme® for the remediation of water saturated porous media polluted with jet fuel (JP-5). Also for comparison purposes, the commercial surfactant sodium dodecyl sulfate (SDS) was used. Bench scale experiments were conducted in a glass column packed with glass beads. The migration of JP-5 in the glass column under various conditions, with and without the presence of GreenZyme® was monitored by a well-established photographic method. Digital photographs of the packed column were captured under fluorescent lighting. The fluorescent intensity of JP-5 dyed with Red Oil O within the column was analyzed using the Matlab Image Processing Toolbox. The colour intensities were converted to concentrations via appropriate calibration curves. The experimental results suggested that GreenZyme® was an efficient biosurfactant capable of enhancing significantly the migration of JP-5 in the glass column, which performed considerably better than SDS under the experimental conditions of this study.

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1. Introduction

Subsurface contamination by non-aqueous phase liquids (NAPLs), as a result of accidental spills of hydrocarbons from leaking tanks, disposal dumps, and pipelines, is a serious and widespread problem, which has been explored by numerous investigators [1–16]. Entrapped or residual light non-aqueous phase liquid (LNAPL) or dense non-aqueous phase liquid (DNAPL) in subsurface formations may exist in the form of ganglia or pools and they are long-lasting sources of contamination [17,18]. The NAPLs can be classified into two major categories: (i) chlorinated industrial solvents (e.g.

trichloroethylene or TCE), and (ii) petroleum fluids (e.g. jet fuel). Understanding the behaviour of NAPLs in the subsurface is essential for the appropriate design of efficient recovery and remediation methodology.

Remediation of NAPL contaminated subsurface formations is traditionally performed by several methodologies including: pump-and-treat, air sparging, bio-remediation, surfactant or bio-surfactant flashing, cosolvent addition, and natural attenuation [19–28], or use of acoustic waves [29–31] could be used for effective mobilization of NAPL ganglia. However, surfactant flashing is one of the most popular methodologies

used because surfactants are delivered into contaminated subsurface formations through the aqueous phase and can provide a relatively fast and economical site remediation [23,26]. Surfactant flashing can effectively enhance NAPL removal from contaminated subsurface formations with both mechanisms of mobilization and solubilization [32].

Jet propellant (JP) fuels are used mainly in military and civilian aircrafts, and consist of relatively complex mixtures of hydrocarbons [33]. Hydrocarbons are composed only of the elements carbon (C) and hydrogen (H) and they are the main constituents of petroleum and natural gas. The hydrocarbons are classified based on their structure into two major groups: aliphatic and aromatic. Aliphatic hydrocarbons are classified based on the types of bonds they contain into: alkanes also called paraffins (contain only single bonds), alkenes also called olefins (contain a carbon-carbon double bond), and alkynes (contain a carbon-carbon triple bond). Aromatic hydrocarbons have unique stability, and they contain one or more planar rings of carbon atoms joined by aromatic (not single) bonds [34]. Alkanes are classified as saturated hydrocarbons (no hydrogen can be added to them), whereas alkenes, alkynes and aromatics as unsaturated hydrocarbons (hydrogen can be added to carbon atoms). The first turbine engines were fueled with plain illuminating kerosine (also spelled as kerosene, which is the general name of paraffin), which was widely available because it was used for wick lamps. Subsequently, after World War II, the U.S. Air Force developed wide-cut jet fuels, also known as naphtha-type (JP-4) from hydrocarbon mixtures produced by distillation of crude oil. Because these fuels were found to have operational disadvantages due to their high volatility, the Air Force in the 1970s developed kerosine-type jet fuels (JP-8). The U.S. Navy used on aircraft carriers a kerosine-type jet fuel (JP-5) since the 1950s. Also, the commercial jet industry developed in the 1950s kerosine-type jet fuels (Jet A, Jet A-1) balancing performance, fuel price and availability, and (Jet B) for its enhanced cold-weather performance [33]. Several other military fuels are available, which have been developed for highly specific applications. Fuel-soluble chemicals are added to some of the kerosine-type jet fuels in very small amounts to enhance fuel performance and fuel handling. These chemicals are referred to as additives. Furthermore, it should be noted that some military fuels are very similar to their civilian counterparts, containing just different additives.

Because JP fuels are flammable as well as potential environmental pollutants, there are numerous regulations available for their distribution and storage. However, jet fuel may be released in the environment in a variety of ways, including on purpose by in-flight

fuel jettisoning (fuel dumping) or accidentally from spills and leaks that can happen during fuel storage, transportation or distribution. In-flight fuel jettisoning happens in certain emergency situations before a return of the plane to the airport shortly after takeoff, or before landing short of its intended destination to reduce the weight of the aircraft. Jet fuel jettisoned from airplanes can be transferred to water or soil via airborne dispersion. Jet fuel spills and leaks happen mainly due to human mistakes (incorrect maintenance, negligence) but also from accidents (pipeline breaks).

The objective of this study was to evaluate the use of inert enzymes that alter the fuel-water-glass dynamics for the remediation of jet fuel from a water-saturated column packed with glass beads, using traditional photographic procedures. To our knowledge, inert enzymes have not been explored before for the remediation of porous media contaminated with jet fuel JP-5.

2. Procedures and materials

2.1. Experimental set up

The experiments were conducted in Chromaflex glass columns with inside diameter of 2.5 cm and length of 30 cm, packed with 2 mm diameter glass beads (Fisher Scientific, New Jersey). The glass beads were carefully cleaned, following the procedures established by Syngouna and Chrysikopoulos [35]. The column was homogeneously packed in the vertical position under standing distilled deionized water (ddH₂O) to minimize the possibility of air entrapment. Screens were placed at the inlet and outlet ends of the column to hold the glass beads in place and to ensure that the inflow was distributed evenly. The screens were secured by column end caps that screw into threads moulded in the ends of the glass column. The porosity of the column was determined as $\theta = 0.42 \pm 0.01$, and the pore volume $PV = 61.5 \pm 1.5$ mL. The packed column was placed horizontally with the aid of strong calipers, on top of a custom made, sandblasted glass covered, light table equipped with a fluorescent light source. Prior to conducting each experiment, the packed column was fully saturated with ddH₂O. Several pore volumes of ddH₂O were flushed through the packed column to remove any air bubbles. Fluids were inserted into the packed column with a peristaltic pump (Masterflex L/S, Cole-Palmer). The jet fuel was injected into the packed column through an injection port mounted at the packed column with a small syringe injector. A 35 mm digital camera (Fujifilm HS20) mounted on a tripod was placed directly above the packed column. A complete schematic illustration of the experimental apparatus is shown in Figure 1.

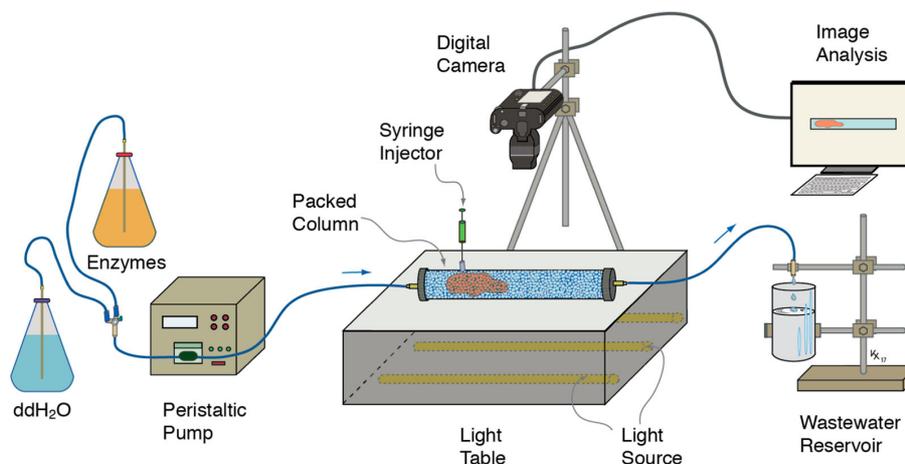


Figure 1. Schematic illustration of the experimental setup.

2.2. Photographic methods and image processing

The photographic and image analysis procedures employed in this work were similar to those reported by Thomas and Chrysikopoulos [36] and Chrysikopoulos et al. [37]. To ensure that all photographs could be directly compared to one another, a few preliminary steps were completed. The camera was set to auto exposure and to natural or standard-red-green-blue (sRGB) colour mode, in order to take a photograph of the most concentrated coloured JP-5 standard. The recorded intensity of the emitted light from the coloured JP-5 was considered to be at the maximum expected level, because all subsequent photographs were expected to have equal or lower recorded intensity. The aperture, shutter speed, and focus settings automatically set by the camera were recorded for use in subsequent photographs taken manually under the same lighting conditions and focus settings.

All sRGB images were stored as three-dimensional (3-D) arrays. Each 3-D array consisted of three two-dimensional (2-D) arrays of pixel red (r_{xy}), green (g_{xy}), and blue (b_{xy}) intensities. Each 2-D array contained the x and y locations (row and column numbers) of the pixels, P_{xy} , in the image, together with the corresponding intensity value, scaled in the range 0–255. These images were easily handled with matrix operations, and observed intensities were correlated to concentrations. In this work, all images were processed with Matlab Image Processing Toolbox [38].

2.3. Jet propellant fuel 5

The jet propellant used in this work is JP-5, which is primary military fuel for land-based military aircraft,

developed by the U.S. Navy. JP-5 is also known by its NATO code F-44, and as Aviation Carrier Turbine fuel (AVCAT fuel). JP-5 is a complex mixture of hydrocarbons, containing 53% paraffins, 31% cycloparaffins, 16% aromatics, 0.5% olefins, and several additives [39]. Some of the physical and chemical properties of JP-5 are listed in Table 1.

2.4. Greenzyme®

Enzymes are generally globular proteins (linear polymers of amino acids), which are synthesized by living cells to work as catalysts [40]. In this study, GreenZyme® was used, which is a water-soluble, DNA–protein-based, biodegradable, non-living enzyme catalyst. GreenZyme® was developed and manufactured by Apollo Separation Technologies Inc., Huston Texas. GreenZyme® is semi-clear yellow, and its odour resembles fermentation. It is completely soluble in water, with specific gravity equal to unity. GreenZyme® is non-flammable, non-hazardous, environmentally friendly with unique abilities to catalyze hydrocarbons, and to detach them from solid surfaces. More specifically, a GreenZyme® water solution gradually diminishes the surface tension between NAPLs and solids, which in turn increases the water-wettability and

Table 1. Physical and chemical properties of JP-5 [39].

Molecular weight	185
Freezing point	−46°C
Boiling point	156–293°C
Flash point (lower flammability temperature)	60°C
Vapor pressure	0.52 mm Hg at 10°C 1.8 mm Hg at 28°C
Specific gravity	0.788–0.834 kg/L at 15°C
Autoignition temperature	246°C

allows previously entrapped NAPL ganglia to migrate in the direction of the interstitial flow. It should be noted that GreenZyme® acts as catalyst so it is not consumed during the NAPL release process. Actually, the enzymes remain either attached on the solid surfaces or dissolved in the aqueous phase. An illustration of the unique GreenZyme® activity and NAPL migration in porous media is shown in Figure 2. It should be noted that GreenZyme® technology has been employed in numerous Enzyme Enhanced Oil Recovery (EOR) applications throughout the world, because it is effective and eco-friendly [41,42].

2.5. Sodium dodecyl sulfate

For comparison purposes, the commercial surfactant sodium dodecyl sulfate (SDS) with linear formula $\text{CH}_3(\text{CH}_2)_{11}\text{OSO}_3\text{Na}$ (Merck, 98,5+%), was also employed in this study. Surfactants accumulate at interfaces because consist of molecules that have both hydrophilic and hydrophobic moieties. Therefore, surfactants are often used as typical detergents or in various soil-remediation processes [43,44]. There are numerous applications of SDS-enhanced remediation of NAPL contaminated subsurface formations [45–47].

3. Calibration curve

In order to correlate the pixel intensities of the pictures to actual JP-5 concentrations in the packed column, a calibration curve was constructed. The pixel intensities were scaled from 0 to 255. A standard solution consisting of 350 mL pure JP-5 coloured with 0.01 gr (0.029 gr/L) red dye (Red Oil O) was used to make by dilution six different JP-5 solutions. The addition of red dye was not expected to alter the physicochemical characteristics of kerosene [48]. The packed column was fully saturated with the standard solution having the lowest percentage

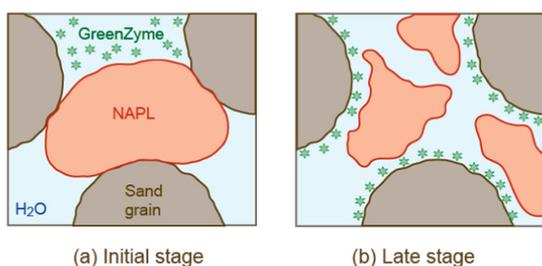


Figure 2. Schematic illustration of GreenZyme® interaction with a NAPL ganglion trapped within a porous formation: (a) initial stage and (b) late stage.

of pure JP-5 (10% of the pure JP-5 standard solution) and a picture was captured. The procedure was repeated with the other five JP-5 dilutions in the order of progressively increasing concentration. For each of the six images captured a modification colour was assigned randomly (see Figure 3). The average of the red, green and blue colour intensities of each picture taken were used to construct the desired calibration curves, which were plots of the averaged three colour intensities versus diluted JP-5 (see Figure 4). The average intensity values shown in the three calibration curves were determined with a simple

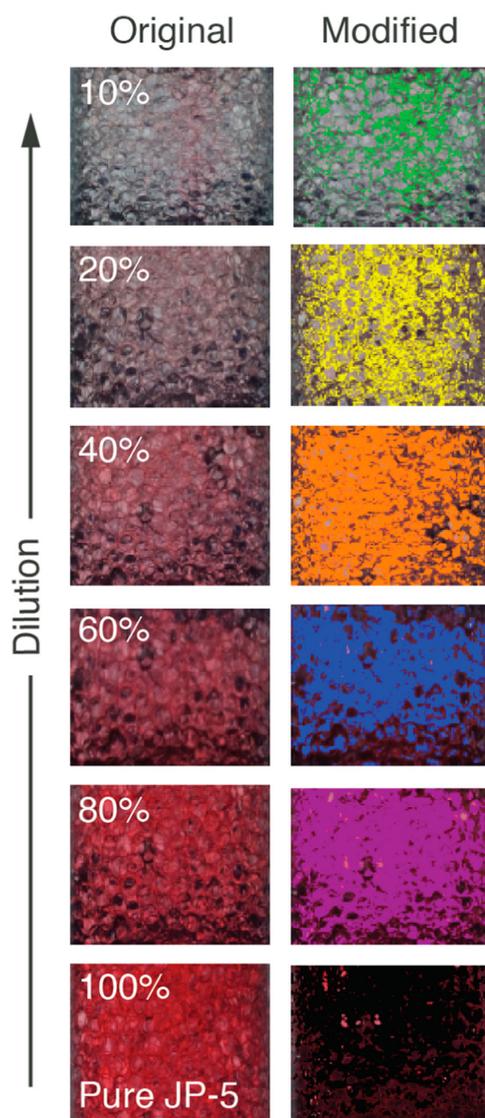


Figure 3. Original and modified pictures of the packed glass column, saturated with various dilutions of pure JP-5 coloured with red dye.

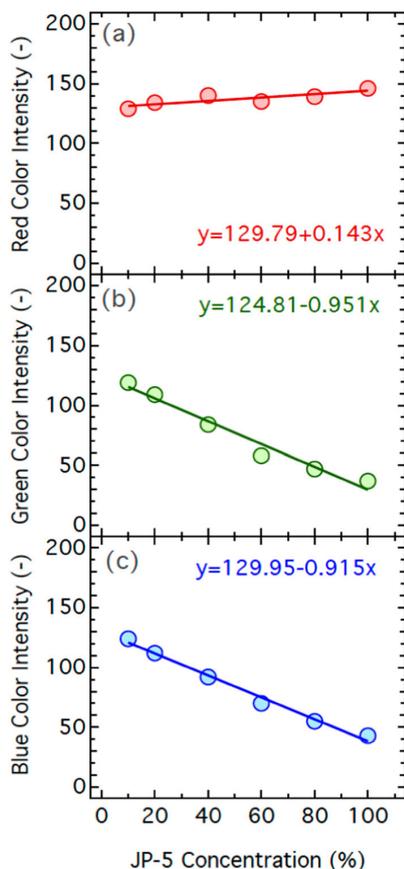


Figure 4. Average of: (a) red, (b) green, and (c) blue colour intensities for various dilutions of pure JP-5.

Matlab algorithm. The algorithm was designed to read the three basic colour (Red, Green, and Blue) intensities, in the sRGB dimensions (0–255), of each and every pixel in a calibration picture and to calculate the corresponding average intensity value of each colour (r_c , g_c , b_c) in the picture.

Photographic image classification (image modification) was conducted with the use of another Matlab algorithm, which was programmed to read the three-colour intensities (r_{xy} , g_{xy} , b_{xy}) of each pixel, P_{xy} , in the image and to calculate the corresponding Euclidean norm, E_n , as follows:

$$E_n = \sqrt{(r_{xy} - r_c)^2 + (g_{xy} - g_c)^2 + (b_{xy} - b_c)^2} \quad (1)$$

Six different E_n values were determined, one for each of the six different dilutions of the pure JP-5 used in this study. Then, the pixel P_{xy} was converted to the colour of the dilution with the calculated smallest E_n value.

4. Results and discussion

4.1. Column experiments

The first experiment was conducted with the water saturated packed column placed in a horizontal position, and was aiming to determine how the movement of JP-5 fuel in water saturated porous media can be enhanced by flashing with water containing GreenZyme®. Water was continuously flashed through the column at a relatively slow flow rate ($Q = 1.0$ mL/min), imitating typical groundwater lows. A 2 mL of dyed JP-5 fuel was injected into the column with a syringe. Very quickly the jet fuel formed a distinct plume within the column. However, after approximately 30 min of water flashing no significant jet fuel migration was observed and the jet fuel plume was practically stabilized. The migration of the jet fuel JP-5 through the column, due to the continuous water flashing, was recorded by taking snapshots. The various pictures were analyzed with the previously described photographic and image analysis procedures. Some of the original and modified images are shown in Figure 5. Subsequently, the column was flashed for 24 h with a water solution containing 5% GreenZyme®. The presence of GreenZyme® enhanced the movement of JP-5 fuel. As shown in Figure 6, the JP-5 plume progressively migrated within the column. To provide a somewhat quantitative analysis of the effects of water flashing compared to GreenZyme® flashing, the JP-5 normalized concentration within the column was averaged over a cross-section just below the injection port. The results were shown in Figure 7(a), where the circles represent measurements over the time period of flashing with ddH₂O, and the squares over the time period of flashing with GreenZyme® solution. Clearly, the presence of GreenZyme® significantly enhanced the movement of JP-5 fuel, and over the experimental duration, a 83% reduction was observed in the normalized JP-5 concentration just below the injection port.

The second experiment was aiming to improve the results of the first experiment by modifying slightly the experimental procedures employed in the first experiment. Specifically, after approximately 30 min of water flashing, when the jet fuel plume remained practically unchanged, 10 mL of a water solution containing 30% GreenZyme® was injected into the column. The flow was stopped, and the GreenZyme® was allowed to remain within the column for a soak period of 48 h. Subsequently, additional 10 mL of a water solution containing 30% GreenZyme® was injected into the column for a second soak period of 48 h. Finally, continuous water flashing of the column was reestablished at a flow rate of 1.0 mL/min. The JP-5 normalized concentrations within the column, averaged over a cross-section just

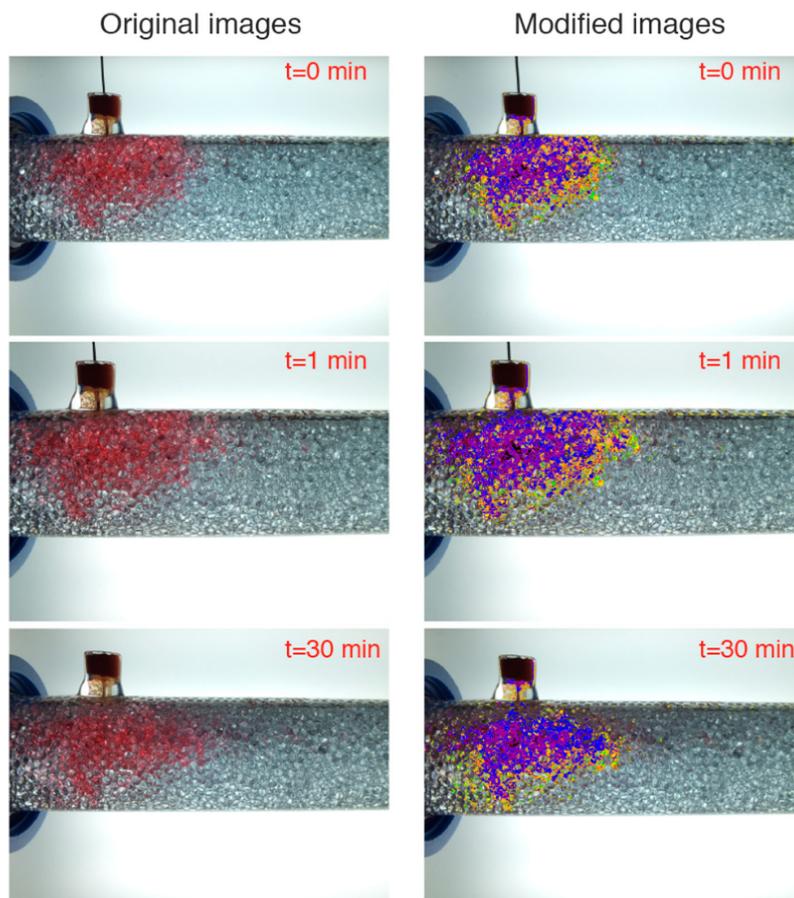


Figure 5. Original and modified images at various times for the distribution of 2 mL JP-5 within the water saturated column flashed continuously with ddH₂O at a flow rate of 1.0 mL/min.

below the injection port, for the various recorded images of the second experiment were shown in Figure 7(b), where the circles represent measurements during flashing with ddH₂O and the diamonds after the soaking with GreenZyme[®] solution. The results suggested that the two periods of soaking with GreenZyme[®] solution in conjunction with water flashing improved the movement of JP-5 fuel, and over the experimental duration a 97% reduction was observed in the normalized JP-5 concentration just below the injection port.

The third experiment was aiming to improve further the migration of JP-5 fuel in water saturated porous media and was conducted by following the exact experimental procedures employed in the second experiment with just one difference. Here, the water temperature of the water flashing of the column just after the two consecutive 48 h soaking periods with 30% GreenZyme[®] solution, was raised from room temperature to 60°C with a water heater. The experimentally determined

JP-5 normalized concentrations within the column, averaged over a cross-section just below the injection port are shown in Figure 7(c), where the circles represent measurements during flashing with ddH₂O prior to the soaks with GreenZyme[®] solution, and the triangles during flashing with heated ddH₂O after the soaks with GreenZyme[®] solution. The results suggested that the two periods of soaking with GreenZyme[®] solution in conjunction with hot water flashing improved the movement of JP-5 fuel, and over the experimental duration a 99% reduction was observed in the normalized JP-5 concentration just below the injection port.

It is worthy to note that in the first experiment smaller JP-5 concentration reduction (83%) was achieved than in the second experiment (97%). In the first experiment 72 mL of stock GreenZyme[®] was utilized over the 24 h flashing with a water solution containing 5% GreenZyme[®], whereas in the second experiment only 6 mL of stock GreenZyme[®] was utilized for the two soaks with

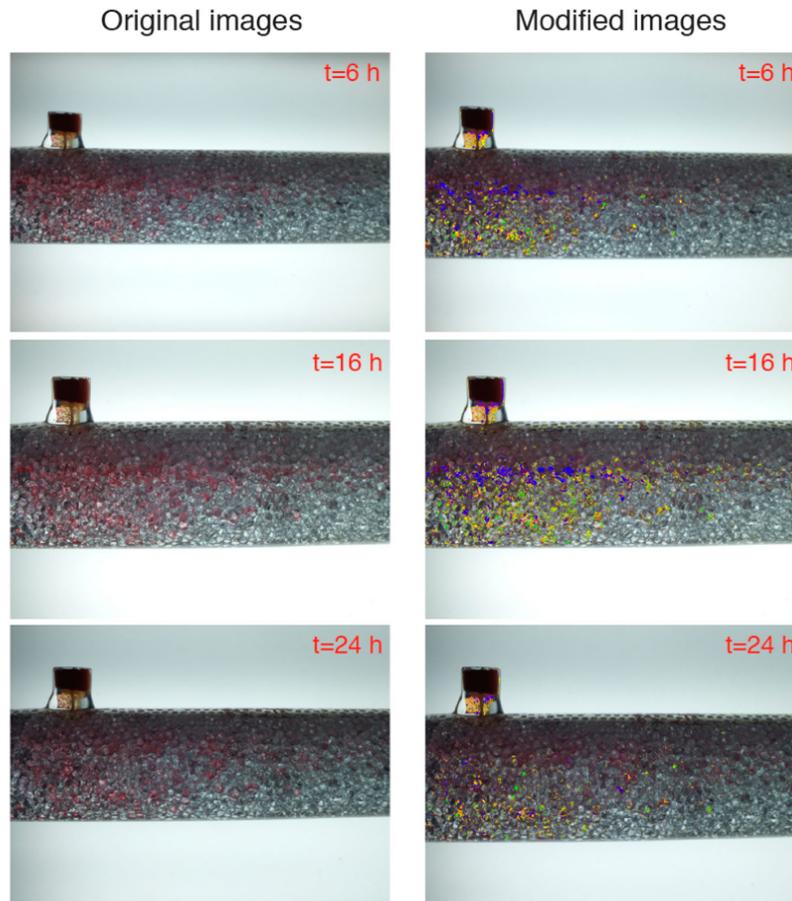


Figure 6. Original and modified images at various times for the distribution of 2 mL JP-5 within the water saturated column flashed continuously with 5% GreenZyme[®] solution at a flow rate of 1.0 mL/min.

30% GreenZyme[®] solution. Furthermore, in the third experiment achieved the best JP-5 concentration reduction (99%) among the three column experiments conducted with GreenZyme[®]. Although only 6 mL of stock GreenZyme[®] was utilized in the third experiment (same as in experiment 2), there was a cost for water heating that should also be accounted for.

For comparison purposes, the fourth experiment was conducted in a fashion similar to the first experiment, but instead of 5% GreenZyme[®] solution the jet fuel in the column was flashed with a 500 mg/L SDS solution, which was gently stirred inside the storage container throughout the experimental duration with a magnetic stirrer (Heidolph). The experimental data of the fourth experiment are shown in Figure 8(a), where the circles represent measurements over the time period of flashing with ddH₂O, and the squares over the time period of flashing with SDS solution at a flow rate of 1.0 mL/min. The results suggested that over the

experimental duration 18% reduction was observed in the normalized JP-5 concentration just below the injection port. Finally, the fifth experiment was practically a repetition of the fourth experiment with just a minor modification, the jet fuel JP-5 in the column was flashed with a 2000 mg/L SDS solution. The experimental data of the fifth experiment are shown in Figure 8(b), where the circles represent measurements over the time period of flashing with ddH₂O, and the diamonds over the time period of flashing with SDS solution at a flow rate of 1.0 mL/min. The results suggested that that over the experimental duration a 74% reduction was observed in the normalized JP-5 concentration just below the injection port. Clearly, the efficiency of flashing with SDS increased with increasing surfactant concentration. However, comparison of Figures 7 and 8 revealed that for the present experimental conditions GreenZyme[®] was more efficient than SDS in enhancing the migration of JP-5 in the glass column.

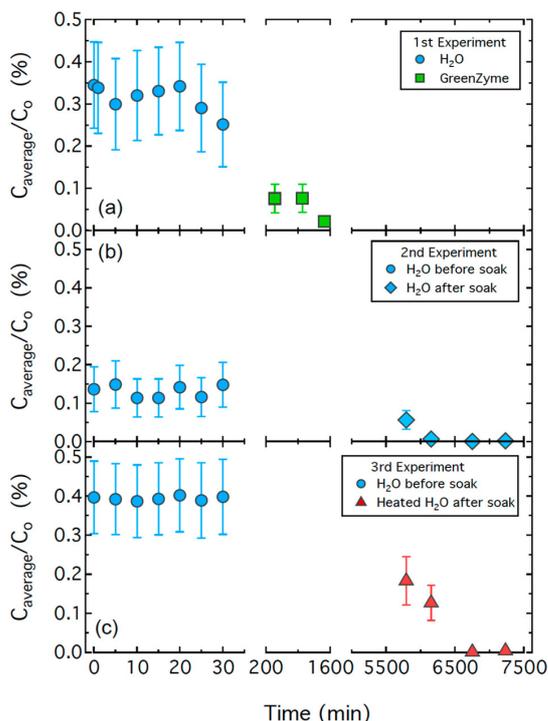


Figure 7. Normalized JP-5 concentrations within the column averaged over a cross-section just below the injection port at various times during: (a) continuous flashing with 5% GreenZyme® solution, (b) two consecutive 48 h soak periods with 30% GreenZyme® solution, and (c) two consecutive 48 h soak periods with 30% GreenZyme® solution and subsequent flashing with 60°C water.

5. Summary and conclusions

This experimental study examined the effectiveness of GreenZyme® for the enhanced removal of jet fuel JP-5 from water saturated porous media. Five experiments were conducted in glass columns packed with glass beads. In three experiments the JP-5 fuel in water saturated porous media was flashed with GreenZyme® solution, and in two experiments with a 500 mg/L SDS solution. The results of various experiments conducted in a column packed with glass beads suggested that GreenZyme® was capable of enhancing the removal of JP-5 within the glass column. It was shown that soaking the column with 30% GreenZyme® solution was more efficient than flashing the column continuously with 5% GreenZyme® solution. Subsequent flashing with water heated at 60°C was shown to be beneficial, but possibly unattractive due to the extra cost. Finally, it was shown that under the conditions of this experimental study, GreenZyme® was more effective

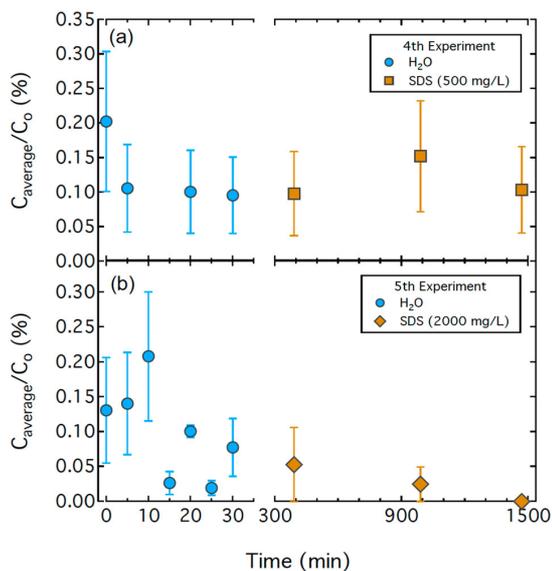


Figure 8. Normalized JP-5 concentrations within the column averaged over a cross-section just below the injection port at various times during: (a) continuous flashing with 500 mg/L SDS solution, and (b) continuous flashing with 2000 mg/L SDS solution.

for enhanced removal of JP-5 in packed columns than the traditional SDS surfactant.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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