

TITLE:

**AUGMENTING A MICROBIAL SELECTIVE PLUGGING TECHNIQUE WITH
POLYMER FLOODING TO INCREASE THE EFFICIENCY OF OIL RECOVERY
- A SEARCH FOR SYNERGY**

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PRINCIPAL AUTHORS:

Lewis R. Brown
Charles U. Pittman, Jr.
F. Leo Lynch

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RECIPIENT:

Mississippi State University
Sponsored Program Administration
P.O. Box 6156

Mississippi State, MS 39762

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TABLE OF CONTENTS

LIST OF TABLES	iv
LIST OF FIGURES	v
ABSTRACT	vi
INTRODUCTION	1
RESULTS AND DISCUSSION	2
Objective.....	2
Results	3
Task 1	3
Task 2	3
Task 3	3
Nuclear Magnetic Resonance Imaging Analysis of Berea Sandstone Cores and North Blowhorn Creek Cores.....	3
Results of NMR Imaging Experiments	5
Investigation of New Polymer (RH-1).....	7
Extensional Viscosity Studies.....	10
Core Flood Experiment to Determine the Effect of Microbial Activity and a Polymer on the Flow Rate of Injection Water	13
Studies Using the Electron Microscope	16
Task 4	22
Task 5	23
Task 6	23
SUMMARY AND CONCLUSIONS.....	24

REFERENCES25

LIST OF TABLES

Table 1. Viscosity measurements on RH-1 (Mwt. $1.25 \cdot 10^6$) Determined at 30.3 °C in standard Injection Brine Solutions12

LIST OF FIGURES

Figure 1. NMR images of cores.....6

Figure 2 One-dimensional T₂ Profile: apparent oil volume per sample length.....8

Figure 3. Core 1 and Core 4 relaxation distribution figures.8

Figure 4. Synthetic equation.9

Figure 5. Flow rate for Berea sandstone core when nutrients are added before polymer14

Figure 6. The oil to water ratio for Berea sandstone treated with nutrients prior to polymer treatment.15

Figure 7. Flow rate for Berea sandstone core when polymer is added before nutrients treatment15

Figure 8. The oil to water ratio for Berea sandstone treated with nutrients prior to polymer treatment16

Figure 9. An electron micrograph of an air-dried sample.....19

Figure 10. Electron micrograph of a sample fixed in 10% gluteraldehyde.....19

Figure 11. Electron micrograph of a sample that has been gluteraldehyde fixed, ethanol and acetone dehydrated, and critical point dried.20

Figure 12. Electron micrograph of a higher magnification of Figure 11.....20

Figure 13. Electron micrograph of a totally disrupted biofilm texture in a sample preserved by dehydration protocol #1.....21

Figure 14. Electron micrograph of a sample preserved by dehydration protocol #3.....21

ABSTRACT

The overall objective of this project is to improve the effectiveness of a microbial selective plugging technique (MPPM) through the use of polymer floods. Of the six tasks to be carried out, one (Task 2) has been completed, two (Tasks 1 and 3) were scheduled to continue, and one (Task 4) was to begin. A new (at this time proprietary) polymer solution has been obtained from Dr. Hester at the University of Southern Mississippi and is being tested in our project.

Sandpack studies have been completed and showed that the injection of both polymer solutions and microbial nutrients caused an alteration of the flow pattern through the pack.

Core flood studies using Berea sandstone are in progress. A second set of core plugs were evaluated by Dr. Watson at Texas A&M and it was found that the problem of oil versus water resolution during magnetic resonance imaging, associated with core flood studies using Berea sandstone could be overcome using D₂O instead of H₂O. Two experimental core plugs from the North Blowhorn Creek Oil Field have been treated and sent to Dr. Watson for evaluation. Core flood studies using Berea sandstone core plugs, prepared to mimic a depleted oil sand, have shown that both polymer flooding and growth of microorganisms in the core plug exhibit reduced flow and result in additional oil recovery.

Electron microscopic examination of some treated Berea core plugs clearly demonstrated that the method of preparation of samples strongly influences the resultant images and can easily lead to misinterpretations.

INTRODUCTION

Over two thirds of all of the oil discovered in this country is still in the ground and cannot be recovered economically with present day technology. Only 27 billion barrels of the approximately 345 billion barrels remaining in known reservoirs is economically recoverable. When primary production becomes uneconomical, secondary and tertiary methods, such as waterflooding, chemical flooding, CO₂ or N₂ flooding, and microbial enhanced oil recovery (MEOR) are employed. Recently, a microbial permeability profile modification (MPPM) procedure was shown to be a cost effective means of enhancing oil recovery.⁽¹⁾ In fact, aside from waterflooding alone, MPPM is the least expensive of the enhanced oil recovery procedures. Since MPPM and permeability modification via polymers are similar in mode of action, it was reasoned that coupling those two technologies might result in a synergy that is not only cost effective but also more efficient in oil recovering.

Dr. Alex Vadie, retired from the University and will no longer be associated with the project. Dr. F. Leo Lynch, a geologist, has been added to the scientific team and J. E. Parker, a retired petroleum engineer has assumed a more prominent role in the core flood work being conducted on the project.

RESULTS AND DISCUSSION

Objective

The overall objective of this project is to improve the effectiveness of a microbial selective plugging technique of improving oil recovery through the use of polymer floods. More specifically, the intent is to increase the total amount of oil recovered and to reduce the cost per barrel of incremental oil.

In order to accomplish these objectives, the following six tasks will be carried out.

Task 1. Select, characterize, and test various polymers for their impact on the microflora indigenous to petroleum reservoirs in terms of their inhibitory capabilities and their biodegradability.

Task 2. Determine the ability of selected polymers to increase the aerial extent (aerial sweep efficiency) of stratal material colonized by microorganisms in sandpacks.

Task 3. Determine the ability of selected polymer flooding protocols in combination with microbial selective plugging techniques to increase oil recovery from Berea sandstone core plugs prepared to mimic a depleted oil sand.

Task 4. Determine the ability of a microbial selective plugging technique in combination with selected polymer flooding protocols to increase oil recovery from live cores obtained from newly drilled wells.

Task 5. Prepare a cost/benefit evaluation of adding a polymer-flooding procedure to a microbial enhanced oil recovery process using a selective plugging technique.

Task 6. Final report preparation.

Results

To facilitate presentation of accomplishments on this project, results will be set forth by task.

Task 1. Select, characterize, and test various polymers for their impact on the microflora indigenous to petroleum reservoirs in terms of their inhibitory capabilities and their biodegradability.

Dr. Hester (University of Southern Mississippi) has supplied us with a polymer solution of a polymer (DOE/NPTO grant) which exhibits very large extensional viscosities relative to their shear-based viscosities. Tests have shown that the polymer is not toxic to most aerobic organisms nor is it degraded by them. Tests using anaerobic cultures are currently underway.

Task 2. Determine the ability of selected polymers to increase the aerial extent (aerial sweep efficiency) of stratal material colonized by microorganisms in sandpacks.

This task has been completed.

Task 3. Determine the ability of selected polymer flooding protocols in combination with microbial selective plugging techniques to increase oil recovery from Berea sandstone core plugs prepared to mimic a depleted oil sand.

Nuclear Magnetic Resonance Imaging Analysis of Berea Sandstone Cores and North Blowhorn

Creek Cores

Working with Dr. Ted Watson (Texas A&M University) we have continued to evaluate the

application of NMR-imaging to observing water flow patterns and oil recovery from cores. Since ^1H -NMR is being used, differentiating water from oil in the cores has been the central problem since both have high hydrogen nuclei content. The first study involved four cores, each prepared as described below.

- Core 1 was a Berea core, initially filled with injection water containing 1.2% wt. EDTA as the manganese salt. This was added to speed up the relaxation times and, hopefully, increase the water oil contrast. Crude oil was pressurized into the core followed by flushing with water (containing EDTA) until no more oil came out.
- Core 2 was a Berea core that was first filled with D_2O , followed by adding oil and then flushing with more D_2O until no more oil emerged. Since no water protons would be present, this experiment was intended to observe only the oil and thus the water's location would be obvious by default.
- Core 3 was treated the same as core 2 but 80% D_2O /20% H_2O was used.
- Core 4 was taken from the North Blowhorn Creek oil field. It was removed from a section of the field which had already undergone water flooding. It was not further treated.

The samples were studied in a GE 2-Tesla CS I-II imager having a 31cm magnet bore, equipped with a 20 G/cm shielded gradient-coil set and birdcage RF coil. Two-dimensional slice images were generated. They provided information on the spatial distribution of the oil. Quantitative one-dimensional images were also obtained and the fluid present was obtained as a function of the

longitudinal direction. Finally, inversion-recovery experiments gave a distribution of T_1 relaxation times in the sample.

Results of NMR Imaging Experiments

1. Relatively little signal was obtained from Core 4 from North Blowhorn Creek. In contrast considerable oil was present in Berea cores 1-3. The oil saturation levels for the four cores were: Core 1, 21.4%; Core 2, 21.9%; Core 3, 24.7% and Core 4, 4.87%. This latter value was actually about 3.7% upon correcting for the lower porosity of the North Blowhorn Creek's core rock. Since Core 2 only can give ^1H absorption resonances for the oil (since D_2O was used instead of H_2O), the agreement among cores 1-3 was excellent, clearly demonstrating that sample 4 had much less oil present.

T_1 inversion-recovery experiments demonstrated that cores 2-4 were quite similar and further supported oil quantitation by this method as being reasonably accurate. The relaxation time in Core 1 was shorter than 0.1 sec. showing that EDTA did, indeed, lower relaxation times.

The level of image resolution was approximately 0.47mm. Example images of these cores can be seen in Figure 1. The dark areas represent rock and the light areas may be attributed to H_2O or oil (in the case of Core 2 only oil would be observed). Examination quickly revealed there were no large water channels through which the water is passing without generally permeating through the cores. Therefore, when water flooding is no longer removing any more oil, the passages through which the water is passing must be generally substantially smaller than 0.4mm in diameter. Thus, water is flowing through small pores which are likely coated with oil on the sides. Many other pores may be completely blocked by oil.

The next step will be to image cores which have been subsequently flooded with polymer solutions and/or subjected to microbial growth conditions by feeding nitrates and phosphates.

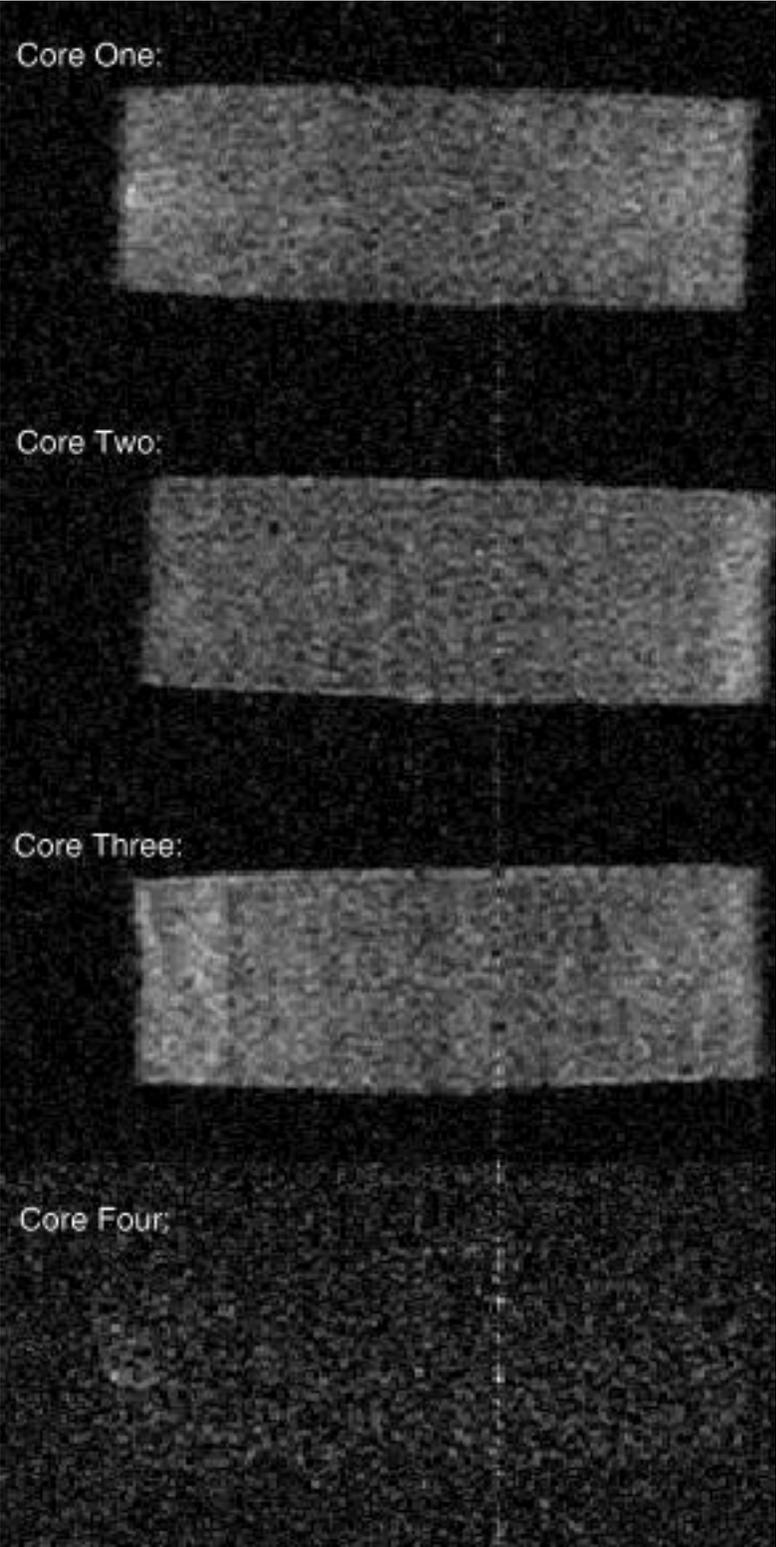


Figure 1. NMR images of cores.

The one-dimensional T_2 profiles for the four cores are shown in Figure 2. These are presented in a format that gives the volume of oil detected per sample length along the length of the cores. Clearly sample Core 4 has far less oil than the other three cores. The reason is that the North Blowhorn Creek core has a substantially lower porosity and permeability than the Berea cores. Another obvious feature observed in this figure is the greater oil depletion at the front end of Cores 1-3 versus the oil present at the back end. The flow of water may have moved some of the oil away from the front end toward the rear. However, this core had been producing only water as pumping continued (e.g. no more oil emerged as water flooding continued).

Figure 3 exhibits the inversion recovery (T_1) relaxation distributions of Core 1 and Core 4. Both of these cores contain water and oil but Core 1 also has EDTA/Mn salt to lower the relaxation time.

Investigation of the New Polymer (RH-1)

A new polymer, RH-1, was synthesized at the University of Southern Mississippi by Professor Roger Hester by the copolymerization of acrylamide and N-acryloyl-3-amino-3-methylbutanoic acid. This polymer has a low molecular weight for a flow control polymer ($MW = 1.25 \cdot 10^6$). See Figure 4. However, it has been chemically designed to have a high extensional viscosity (described more in the following section).

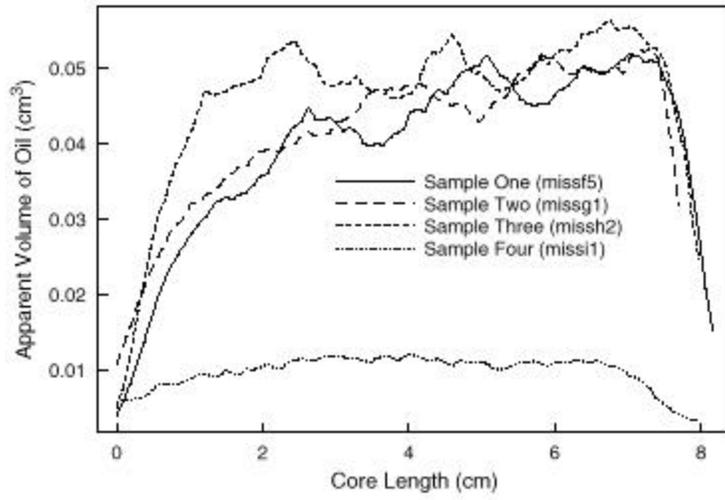


Figure 2. One-dimensional T_2 Profile: apparent oil volume per sample length.

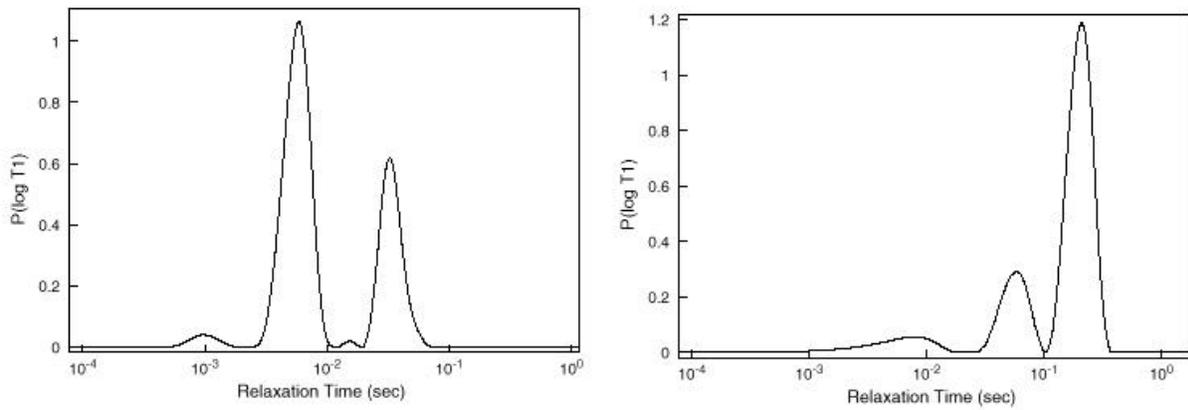
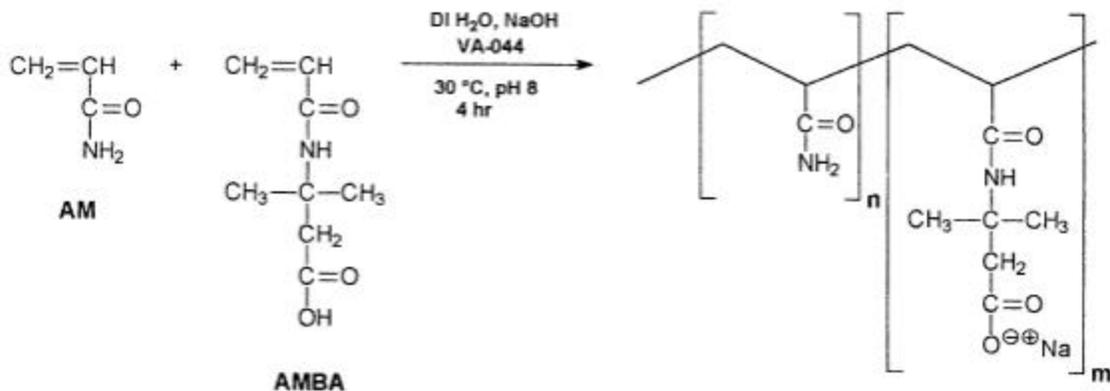


Figure 3. Core 1 and Core 4 relaxation distribution figures.



- Synthetic parameters:
 - $[\text{M}]_{\text{total}} = 0.40\text{ M}$
 - $[\text{M}]:[\text{I}] = 2000$ (0.05 mol% based on $[\text{M}]_{\text{total}}$)
 - NaAMB-10-2: 10 mol% AMBA in feed
 - Mol. wt. 1.25 MM (e.g. 1.25×10^6)

Figure 4. Synthetic equation.

This polymer was tested by pumping it through cores which had been flooded with standard injection water until no more oil would come out. The new RH-1 polymer effectively removed more oil. This is a significant result given the low molecular weight of this polymer which was also used at a low (500 ppm) concentration. Low molecular weight polymers give only modest enhancements in the shear viscosity. This suggests that the critical extension viscosity of RH-1 may have played a role in the removal of more oil from the core.

Core A This core was saturated with oil (while water wet) at a pressure drop of 190psi

(250psi inlet with 60psi back pressure). Eighteen ml of water was taken up in the core. Then 395ml of injection water was pumped through at an inlet pressure of 200psi and a back pressure of 60psi. Nine ml of oil was collected before no more oil could be removed. Thus, water flooding removed 9 of the 18ml of oil in the core. Subsequent flooding with 385ml of a 500ppm solution of RH-1, at an injection pressure of 300psi and back pressure of 60psi, removed an additional 2ml of oil (> 20% recovery of the remaining oil). The flow rate of injection water vs 500ppm of RH-1 in injection water through the core is instructive. The polymer did not slow

	P	FLOW RATE
Injection Water (start)	140psi	2.54 ml/min
500ppm RH-1 injection water	240psi	5.17 ml/min
Injection Water (after)	40psi	6.67 ml/min

down the flow through the core appreciably (6.67 ml/min for injection water vs 5.17 ml/min for injection water plus 500ppm of RH-1).

Another core was treated in a similar fashion. Water flooding alone removed 9ml of oil and polymer flooding (500ppm RH-1) 1ml more of oil. This core only took up 11 to 12ml of oil initially. The polymer flow rate was 0.81 ml/min (P=290psi) while the injection water flow rate was 1.05 ml/min (P=290psi).

Extensional Viscosity Studies

Studies have just started on determining the extensional viscosities of polymers used for flow

control. The calculation of the approximate extension rate, v_c , for a fluid which is being forced through a porous medium (strata or core) is a very important consideration. When the fluid's extension rate equals the critical extension rate of the polymer coil, v_c , the polymer coil will rapidly unfold or unwind and extend in the direction of flow. This event causes energy to be absorbed. Therefore, when the polymer coil rapidly extends, the fluid flow resistance through the porous media will go up sharply. When this occurs flow control will increase. In other words, at the critical extension rate the fluid's effective viscosity goes up, helping divert the fluid in another flow direction where the rate of flow is slower. The fluid extension rate is described by:

$$v_c = 2^{1/2} / \sqrt{\phi} (v/d)$$

Where v = fluid extension rate
 ϕ = porosity of media (core)
 d = effective sand sphere diameter of porous media
 v = velocity of flow through the porous media

v_c = value of v where the polymer elongates

The critical extension rate of a polymer can be expressed as:

$$v_c = (6 \sqrt{RT}) / (25 M \eta_0 / \mu)$$

Where M = molecular weight
 R = gas law constant
 T = temperature in K
 η_0 = intrinsic viscosity of polymer
 μ = shear viscosity of the fluid

Since polymer 1285 from Alcoflood has been used in previous experiments, its critical extensional velocity will be studied. Also, the special polymer RH-1 will be investigated because its structure has been designed to give a large extension effect. Solutions of these and other polymers will be pumped at various flow rates through well characterized Berea cores (known porosity, ϕ from the

uptake experiments and permeability which will be determined according to $k=QL\mu/S \Delta P$ where Q =volumetric flow rate through a porous media of length L and cross sectional area S of a Newtonian fluid having a shear viscosity of μ , where ΔP is the pressure drop across length L). The intrinsic viscosities in the standard injection solution will be determined by dilution viscometry. The polymer solutions of a known concentration will be pumped through cores at a series of different pressure drops (e.g. a series of flow rates). Plots of the flow rate versus ΔP will be linear up to the point the fluid's extension velocity reaches the polymer's critical extension velocity. A change in slope will indicate the point $= v_c$.

The viscosity behavior of RH-1 was studied in the standard injection water. These data are summarized in the table below. The purpose of these data are, not only to characterize this polymer but also, to eventually see if the polymer adsorbs to the core and to determine if shear degradation of RH-1 occurs when it is pumped through Berea cores. We expect that less shear degradation should occur since its molecular weight and coil hydrodynamic volume is much lower than other mobility control polymers we have studied. However, these measurements have not yet been made. Viscosity measurements will be made after pumping a polymer solution through a Berea core to establish whether or not adsorption is occurring and to look for shear degradation at high and low flow rates through the core.

Table 1. Viscosity measurements on RH-1 (Mwt. $1.25 \cdot 10^6$) Determined at 30.3 °C in standard Injection Brine Solutions.

Concentration (g/100cc)	t_o (sec.)	$t_{(soln)}$ (sec.)	t/t_o	n_{sp}^a	n_{inh}^a	n_r^a
0.1000	101.6	350.5	3.45	2.45	0.896	2.45

0.0833	101.6	338.4	3.33	2.33	1.016	2.80
0.0714	101.6	295.5	2.91	1.91	0.906	2.67
0.0625	101.6	260.8	2.57	1.57	0.720	2.51
0.0556	101.6	238.8	2.35	1.35	0.541	2.43
0.0500	101.6	227.1	2.24	1.24	0.423	2.47

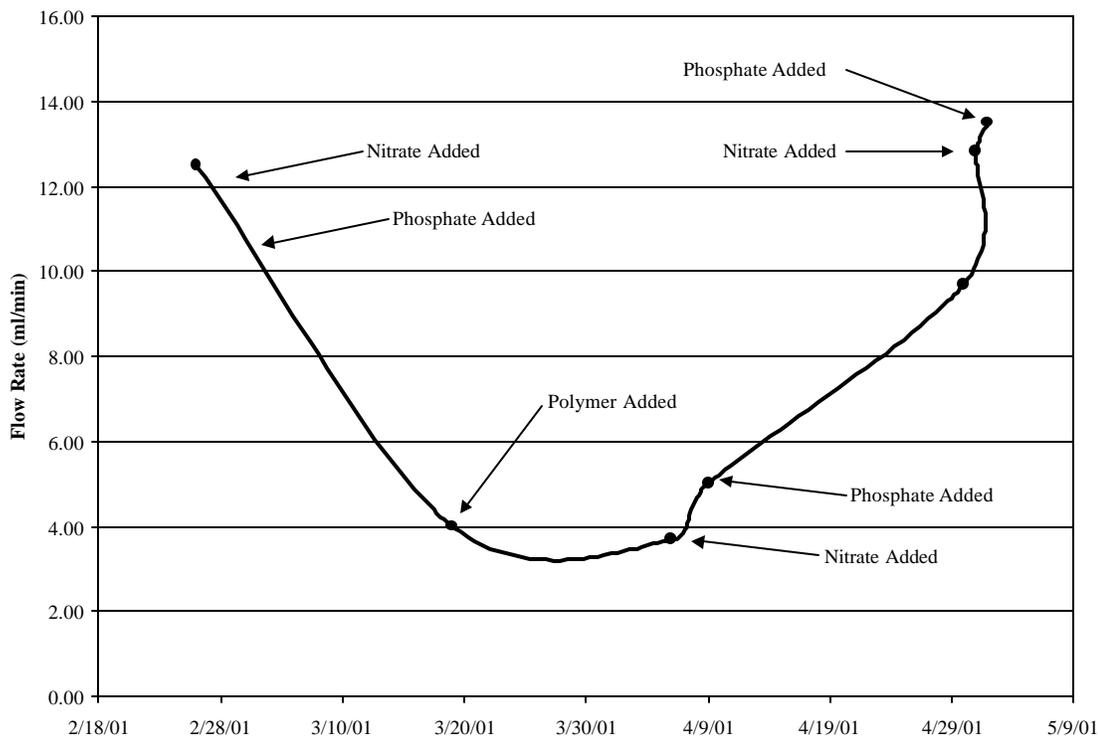
$$^a n_{sp} = t/t_o - 1; n_{inh} = (1/c)\ln(t/t_o); n_r = n_{sp}/\text{conc.}$$

Core Flood Experiment to Determine the Effect of Microbial Activity and a
Polymer on the Flow Rate of Injection Water

Prior to the start of these experiments, Berea sandstone cores were prepared so that they represented a depleted oil formation. In order to simulate a depleted oil formation, the Berea cores were evacuated to replace the voids in the core with injection water and microbial cells. These saturated cores were placed into the Hassler sleeves and oil pumped through the cores until no more water was present in the effluent. Following this treatment, injection water was pumped through the core until no more oil was present in the effluent. At this point the cores were considered to be representative of a depleted oil formation.

The cores representing a depleted oil formation were employed in experiments conducted to determine the effects of microbial activity combined with polymer flooding on the flow rate of injection water through these cores. When microbial activity is encouraged through the addition of NaNO_3 and Na_2HPO_4 and then followed by an application of polymer, the flow rate is greatly reduced (see Figure 5). This suggests that the flow paths established prior to the start of the investigation were beginning to become plugged. Figure 6 offers further evidence that established flow paths are being plugged and new flow paths are being established by the presence of oil 61 d after treatment began. The impact on

flow rates, as shown in Figure 7, was more pronounced when the core was treated first with polymer followed by the addition of nutrients to support microbial activity. The flow rate decreased from 9.5 ml/min to 0.8 ml/min with the addition of the polymer solution. The flow rates slowly increased to 8.0 ml/min with the addition of the nutrient solutions suggesting that the polymer was being removed from the flow paths. The flow rate was decreased again 5 d later suggesting that microbial growth was responsible for the decrease. Evidence for the plugging of the established flow paths and the



establishment of new flow paths may be seen in Figure 8 with the presence of oil 37 d after treatment began. The quicker response by the microorganisms when the core was treated first with polymer then nutrients may suggest that the order in which polymer treatment is applied may have a significance impact on flow rates. Further experimentation will determine if the order in which treatment is applied

has an effect on the enhancement of microbial activity.

Figure 5. Flow rate for Berea sandstone core when nutrients are added before polymer.

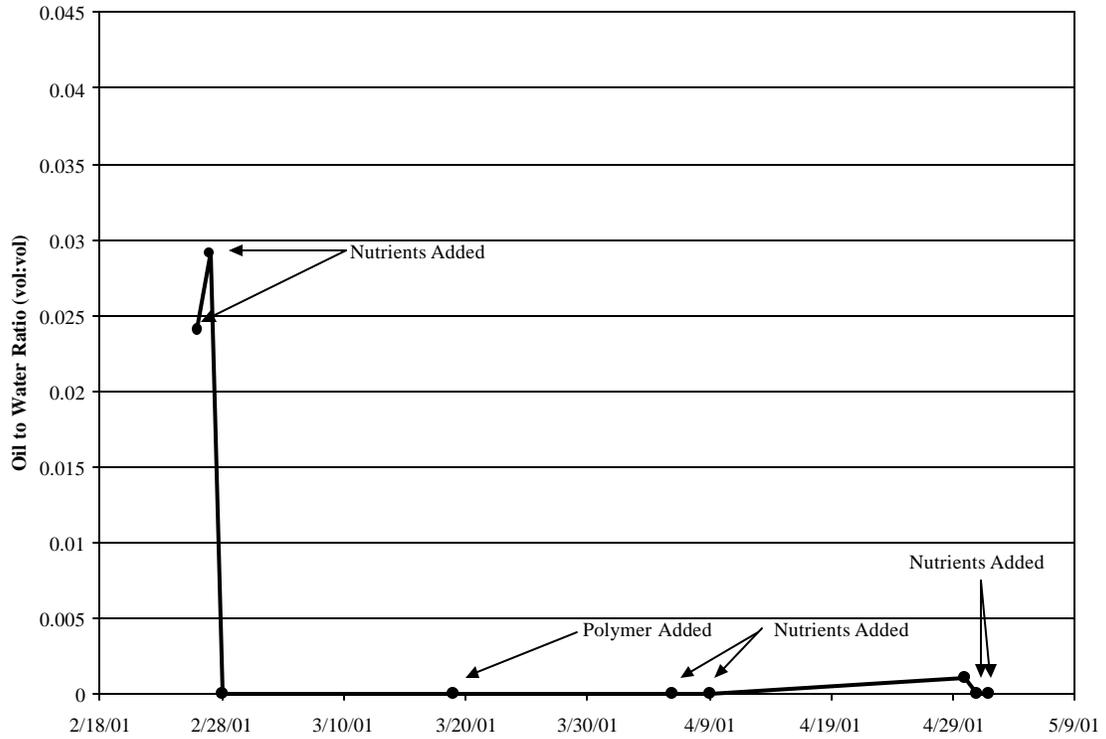
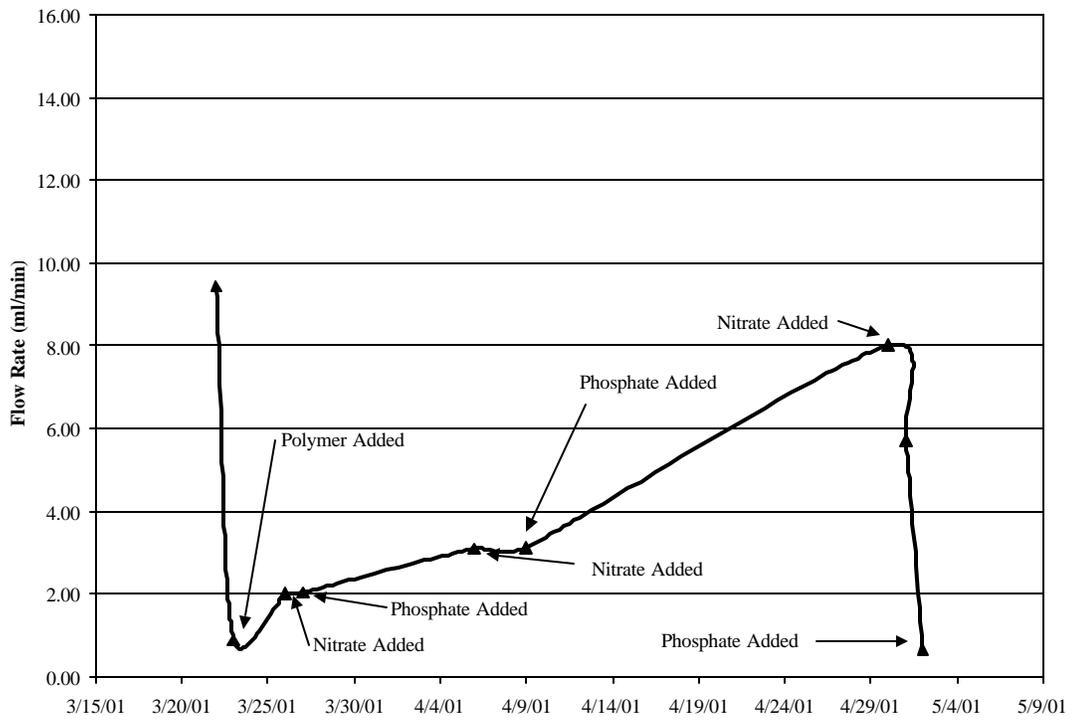


Figure 6. The oil to water ratio for Berea sandstone treated with nutrients prior to polymer treatment.
 Figure 7. Flow rate for Berea sandstone core when polymer is added before nutrients.



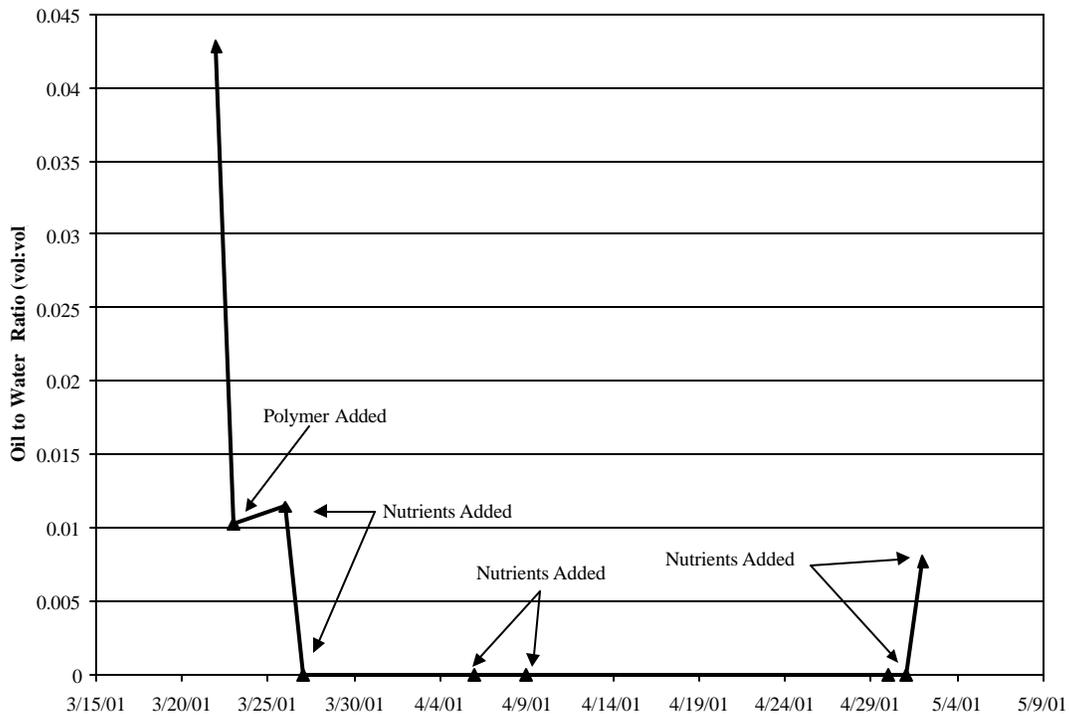


Figure 8. The oil to water ratio for Berea sandstone treated with nutrients prior to polymer treatment.

Studies Using the Electron Microscope

An understanding of the spatial relationships between organic matter (bacteria and biofilm) and the mineral matrix and porosity in a rock is necessary to fully understand the MEOR process. Sample preparation and imaging procedures for confocal and scanning electron microscope (SEM) are being developed to investigate these relationships. Rock samples have been inoculated with bacteria and fed, and have then been prepared for SEM analysis using several widely accepted methods that are purported to preserve organic textures. The purpose of these experiments is to contrast and evaluate these techniques in order to determine which procedure best preserves the *in situ* bacterial and

microfilm textures and the relationships between these organic materials and the porosity and rock matrix.

The organic-preservation protocols that have been tested are:

1. Gluteraldehyde fixation and ethanol dehydration:

Step 1. Primary fixation: fix specimens in 2.5% gluteraldehyde for 2 hours.

Step 2. Dehydrate in ethanol, 10-15 min. per step: 35%, 50%, 70%, and 95% ethanol.

Step. 3. Continue dehydration: 100% ethanol (4 changes) for one hour.

Step 4. Change to HMDS (hexamethyldisilazane), 2 changes, 10 minutes each.

Step 5. Air dry sample.

2. Gluteraldehyde fixation, ethanol dehydration, and critical-point drying.

Step 1. Primary fixation: fix specimens in 2.5% gluteraldehyde for 2 hours.

Step 2. Dehydrate in ethanol, 10-15 min. per step: 35%, 50%, 70%, and 95% ethanol.

Step. 3. Continue dehydration: 100% ethanol (4 changes) for one hour.

Step 4. 5 CO₂ flushes in Polaron Critical Point Drier.

3. Gluteraldehyde fixation, ethanol dehydration, acetone dehydration, and critical-point drying.

Step 1. Primary fixation: fix specimens in 2.5% gluteraldehyde for 2 hours.

Step 2. Dehydrate in ethanol, 10-15 min. per step: 35%, 50%, 70%, and 95% ethanol.

Step. 3. Continue dehydration: 100% ethanol (4 changes) for one hour.

Step 4. Dehydrate in acetone, 10-15 min per step: 35%, 50%, 70%, and 95% acetone.

Step 5. 5 CO₂ flushes in Polaron Critical Point Drier.

4. Gluteraldehyde fixation. Fix sample in 10% gluteraldehyde for 2 hours.
5. Air dry.

Figure 9 is an example of a sample that has been air dried. Biofilm slime covers both the rock matrix and the cylindrical bacteria. Figure 10 (sample fixed in 10% gluteraldehyde) shows similar features: biofilm slime (S) occurs as a more or less continuous coat over both rock and bacteria (B).

Figure 11 shows the textures present in a sample that was preserved according to protocol #3 (gluteraldehyde fixation, ethanol and acetone dehydration, and critical point drying). In this sample the biofilm slime is dessicated and curled. A higher-magnification image of this sample (Figure 12) shows that this dehydration and drying procedure completely changes the morphology of the biofilm from a continuous coat (Figures 9 & 10) to tattered remnant sheets and strings. This procedure does, however, better preserve the texture of the bacterial cells themselves than does simple air drying or gluteraldehyde fixation (Figures 9 & 10).

Figure 13 shows a totally disrupted biofilm texture in a sample preserved by dehydration protocol #1. The arrows point to remnant biofilm fragments that very closely resemble small bacteria. Figure 14 (sample preserved by dehydration protocol #3) is a high magnification image of comparable features. Similar <100 nm features have been interpreted as nannobacteria or ultramicrobacteria in terrestrial and extraterrestrial geologic samples.

The tentative conclusions from these sample preservation experiments is that no tested

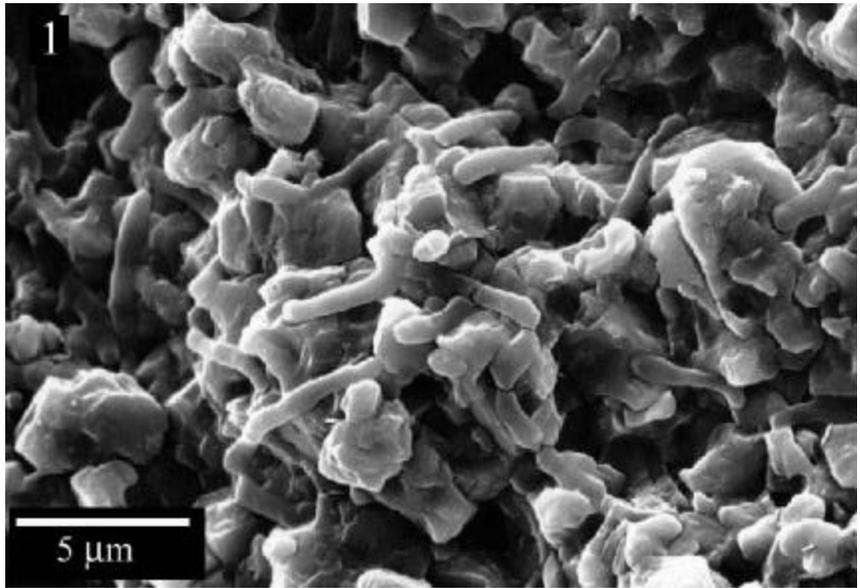


Figure 9. An electron micrograph of an air-dried sample.

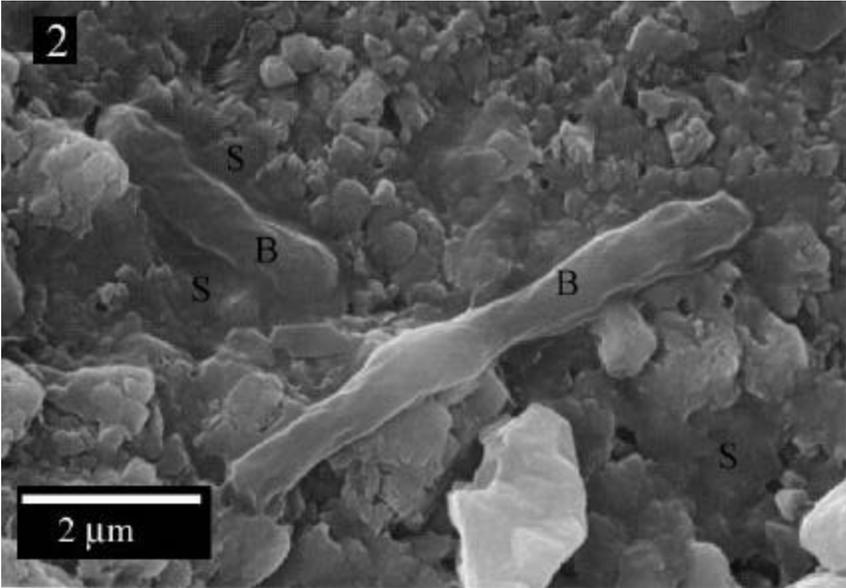


Figure 10. Electron micrograph of a sample fixed in 10% gluteraldehyde.

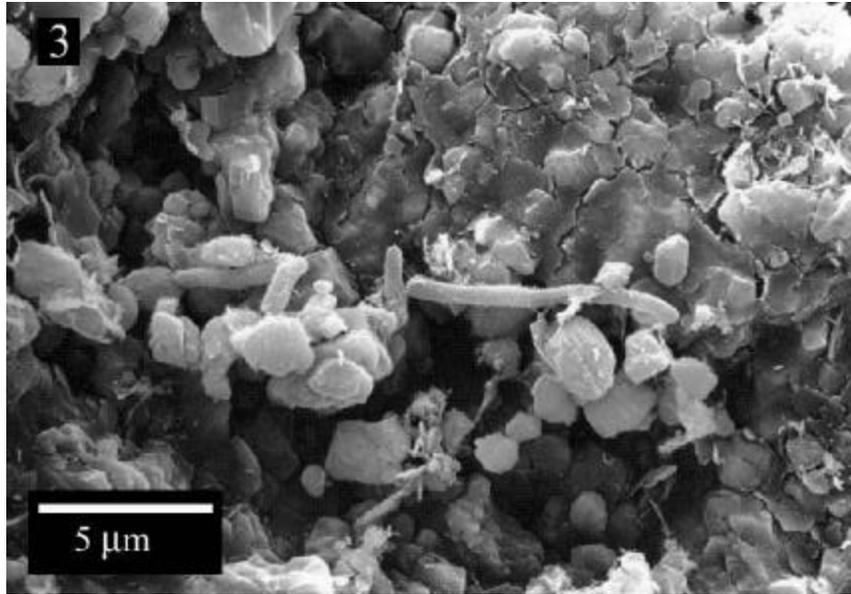


Figure 11. Electron micrograph of a sample that has been glutaraldehyde fixed, ethanol and acetone dehydrated, and critical point dried.

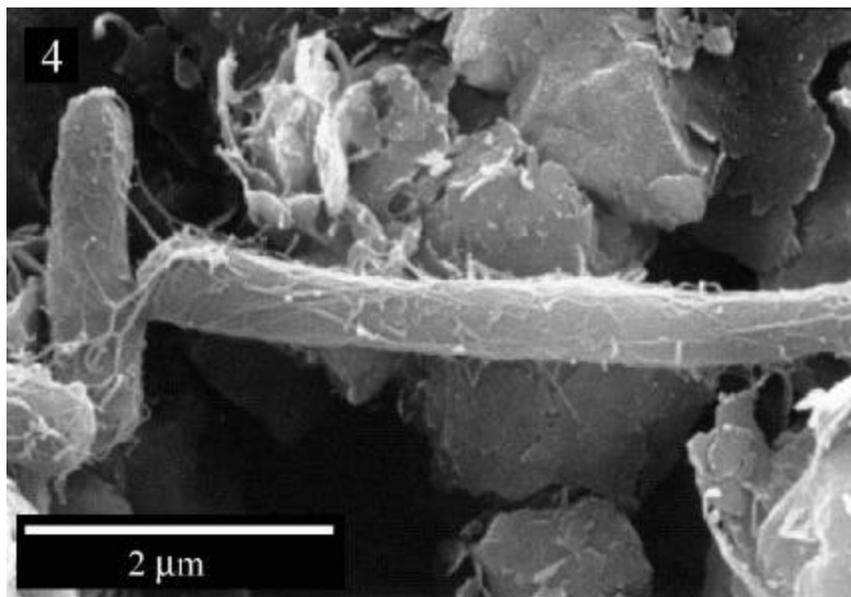


Figure 12. Electron micrograph of a higher magnification of Figure 11.

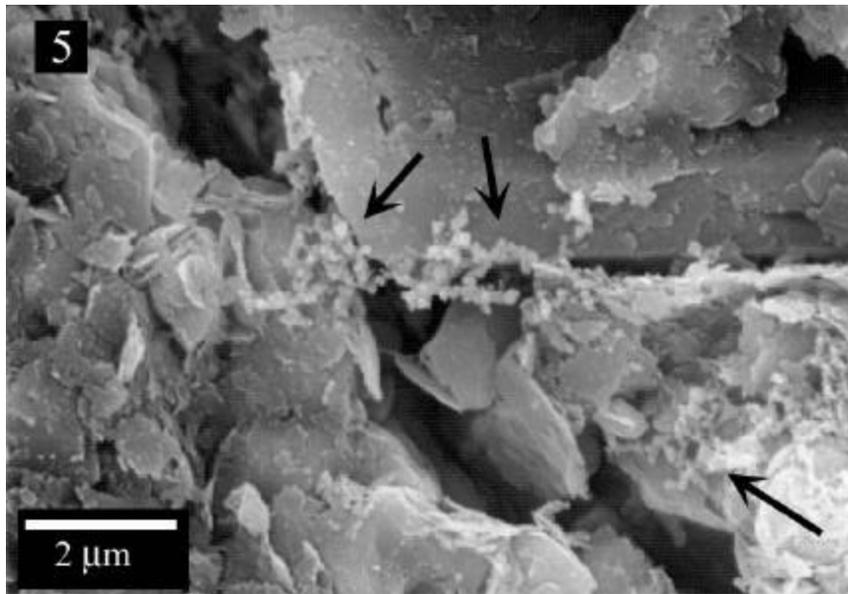


Figure 13. Electron micrograph of a totally disrupted biofilm texture in a sample preserved by dehydration protocol #1.

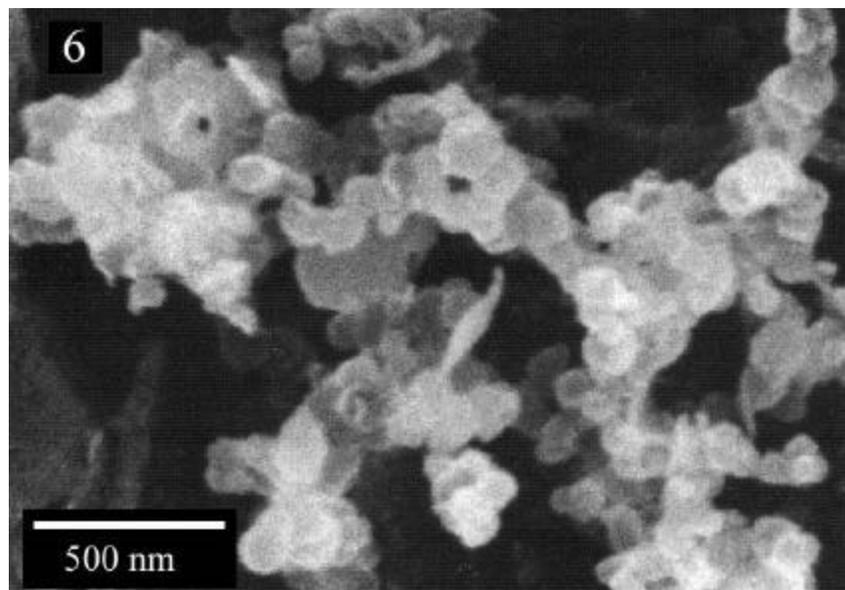


Figure 14. Electron micrograph of a sample preserved by dehydration protocol #3.

protocol correctly maintains the *in situ* textures of both the bacteria and the biofilms. Air drying or simple gluteraldehyde fixation best preserves the texture of the biofilm; however, bacterial bodies are distorted. In contrast, bacterial bodies are well preserved by any of the dehydration techniques; however, these techniques greatly distort and corrupt the texture of the biofilm. Furthermore, the dehydration techniques produce textures in the remnant biofilm that bear a strong resemblance to small biological entities, commonly referred to as nannobacteria. Experiments on different bacteria and biofilms are in progress.

Larger scale organic/inorganic relationships will be studied using standard petrographic and confocal microscopy. Different fluorescent stains (such as cell wall, live vs. dead, biofilm, etc.) are being tested on samples for conoscopic study. Ultra-thin flat samples will be produced by low-viscosity vacuum impregnation and diamond microtoming. These samples will also be studied using the confocal microscope.

Task 4. Determine the ability of microbial selective plugging technique in combination with selected polymer flooding protocols to increase oil recovery from live cores obtained from newly drilled wells.

Work on this phase of the project has been delayed somewhat due to the highly encouraging opportunity of making these experiments significantly more meaningful by the potential results from the core analyses being designed in concert with Dr. Watson of Texas A&M.

Attempts to obtain new cores from recently drilled wells have not been successful but cores

from previously drilled wells have proved satisfactory since they have been preserved under nitrogen.

These cores are in as close to their original state as possible and do contain indigenous microorganisms in their native state.

Task 5. Prepare a cost/benefit evaluation of adding a polymer-flooding procedure to a microbial enhanced oil recovery process using a selective plugging technique.

Not scheduled.

Task 6. Final report preparation.

Not scheduled.

SUMMARY AND CONCLUSIONS

A new (at this time proprietary) polymer solution has been obtained from Dr. Hester at the University of Southern Mississippi and preliminary studies on its use in this project has begun. It is not toxic to aerobic cultures nor is it degraded by them. Tests with anaerobic cultures are presently underway.

Sandpack studies have been completed and showed that the injection of both polymer solutions and microbial nutrients caused an alteration of the flow pattern through the pack.

Core flood studies using Berea sandstone are in progress. A second set of core plugs were evaluated by Dr. Watson at Texas A&M and it was found that the oil versus water resolution problem associated with core flood studies using Berea sandstone could be overcome using D₂O instead of H₂O. Two experimental core plugs from the North Blowhorn Creek Oil Field have been treated and sent to Dr. Watson for evaluation. Core flood studies using Berea sandstone core plugs prepared to mimic a depleted oil sand have shown that both polymer and growth of microorganisms in the core plug exhibit reduced flow and result in additional oil recovery.

Electron microscopic examination of some treated Berea core plugs clearly demonstrated that the method of preparation of samples strongly influences the resultant images and can easily lead to misinterpretations.

REFERENCES

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