NIPER/BDM-0132

Final Report

LABORATORY FEASIBILITY STUDY OF ASP SYSTEMS FOR THE KARAMAY RESERVOIR

Phase 2
Oil Recovery Evaluations

by
Bonnie L. Gall, Ph. D.

April 1995

Work Performed Under Contract No.
DE-AC22-94PC91008

Prepared for
INTRATECH, Inc.

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BDM-Oklahoma, Inc.
P.O. Box 2565
Bartlesville, Oklahoma 74005

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LABORATORY FEASIBILITY STUDY OF ASP SYSTEMS FOR THE KARAMAY RESERVOIR
Phase 2 Report

EXECUTIVE SUMMARY

Phase 2 of a laboratory feasibility study of alkaline-surfactant-polymer (ASP) systems for the Karamay reservoir was conducted at BDMs Oklahoma/The National Institute for Petroleum and Energy Research (NIPER) for Intratech, Inc. The studies included evaluation of Karamay oil recovery potential of several promising ASP systems using Berea sandstone cores and either consolidated or crushed (sandpack) rock samples form the Karamay reservoir. This report also describes formation damage studies to determine the effect of different salinity brines on Karamay reservoir permeability, static and dynamic adsorption studies, and a steady-state relative permeability for one Karamay reservoir core plug.

Alkaline-surfactant systems have been designed to result in low interfacial tension (≈ 0.001 mN/m) with Karamay reservoir oil over a fairly broad range of salinity and alkalinity conditions. The most effective alkaline agent was 1.6 wt % sodium carbonate. Both Petrostep B-100 and the Chinese surfactant CJ2 were effective surfactants; however, CJ2 was preferred as it is readily available at the reservoir location (in China). For the Berea sandstone core, surfactant concentrations of 1% or higher were required to produce more oil than with an alkaline-polymer (AP) slug only with the Karamay reservoir core, the alkaline-surfactant-polymer (ASP) formulations were more successful than AP slugs.

Karamay reservoir core initially saturated with a non damaging brine (3% KCl) was reduced in permeability after pumping Karamay formation water (TDS ≈ 7000 ppm). The permeability reduction was approximately 50%. Subsequent pumping of Karamay river water (TDS ≈ 340 ppm) resulted in a ten-fold reduction in permeability. The sensitivity of reservoir rock was tested with different concentrations of NaCl brine. Permeability appeared to remain stable for NaCl concentrations greater than 2%. Permeability declined slowly when 1.5% NaCl was used. The results indicate that problems can be anticipated if too low salinity brine is introduced into the Karamay reservoir as part of an ASP project.

ASP chemical system can be adjusted to higher salinities by addition of cosurfactant (ethoxylated nonionic and anionic surfactant) to improve surfactant solubility. Systems were tested at 2.5% Na_2CO_3. Additional evaluations are required to optimize systems at higher salinity

Chemical adsorption was high in several tests with the Karamay reservoir rock due to the high clay content. Alkali consumption was on the order of 0.6-1.5 mg/g for static tests and 0.03 to 0.3 mg/g in dynamic tests. Polymer adsorption was approximately 0.12 mg/g in static tests; and surfactant loss exceeded 1 mg/g in static tests and dynamic tests using crushed reservoir rock. In the field, chemical losses could be significant. Better characterization of the reservoir rock would improve the prediction of chemical losses during ASP flooding.

A summary of results and recommendations for selecting an ASP formulation are provided.

INTRODUCTION

BDM-Oklahoma/National Institute for Petroleum and Energy Research (NIPER) conducted a project entitled "Laboratory Feasibility Study of an ASP System for the Karamay Reservoir" under contract number 7704-000 between U.S. Department of Energy (DOE) and Intratech, Inc.

Alkaline surfactant polymer (ASP) method has been demonstrated as an effective oil recovery technology.[1] It uses low-cost alkali to saponify the natural organic acids in the crude oil to generate surfactants. Adding dilute concentrations of a synthetic surfactant enhances the interfacial activities of the system and also adds flexibility by increasing the optimal salinity range. Research has demonstrated that using lower pH alkalis such as $NaHCO_3$ and Na_2CO_3 is as effective as high pH alkalis (hydroxides and silicates), and also reduces alkali consumption and scale formation.

Phase 1 of the ASP feasibility study for the Karamay reservoir, several alkali and surfactant systems were screened to determine candidate chemical systems to produce incremental oil. Interfacial tension measurements indicated that several surfactants, including Petrostep B-100 and KPS-CJ2, exhibited favorable behavior with Karamay oil, and the most likely alkali candidate for the Karamay reservoir was sodium carbonate. Adding polymer to alkali-surfactant formulations did not significantly degrade interfacial properties. These studies are summarized in the phase 1 status report.

Alkali consumption by Karamay reservoir core was shown to be relatively low. Initial static measurements of surfactant adsorption were high, however. In addition, cation exchange capacity measurements were high, indicating that reservoir rock clay content was high. Additional adsorption measurements have been conducted under both static and dynamic conditions, and the results are summarized in this report.

Phase 2 of the project included a laboratory coreflood study to determine:

1. Formation damage potential in Karamay reservoir core caused by injecting different salinity brines
2. Karamay oil recovery in Berea sandstone for several chemical systems, including those adjusted for different levels of salinity
3. Karamay oil recovery in reservoir rock using the most promising chemical systems

Additional studies included determining a steady-state relative permeability curve for one Karamay reservoir core plug and using CT-imaging to monitor oil saturation during ASP oil recovery experiments.

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[1] French, T. R. and T. E. Burchfield. Design and Optimization of Alkaline Formulations SPE/DOE paper 20239 presented at the SPE/DOE Symposium on EOR, Tulsa, OK, April 22-25, 1990.

RESULTS

Introduction

In Phase 1 of the ASP feasibility study for the Karamay reservoir, several alkali and surfactant systems were screened to determine candidate chemical systems to produce incremental oil. Interfacial tension measurements indicated that several surfactants, including Petrostep B-100 and KPS-CJ2, exhibited favorable behavior with Karamay oil, and the most likely alkali candidate for the Karamay reservoir was sodium carbonate. Adding polymer to alkali-surfactant formulations did not significantly degrade interfacial properties. These studies are summarized in the phase 1 status report.

Alkali consumption by Karamay reservoir core was shown to be relatively low. Initial static measurements of surfactant adsorption were high, however. In addition, cation exchange capacity measurements were high, indicating that reservoir rock clay content was high. Additional adsorption measurements have been conducted under both static and dynamic conditions, and the results are summarized in this report.

Based on these results, a laboratory coreflood study was designed to determine:

1. Formation damage potential in Karamay reservoir core caused by injecting different salinity brines
2. Karamay oil recovery in Berea sandstone for several chemical systems, including those adjusted for different levels of salinity
3. Karamay oil recovery in reservoir rock using the most promising chemical systems

Results obtained and problems encountered during the course of the study resulted in continuing adjustment and evaluation of a chemical system for the Karamay reservoir.

Materials

Table 1 lists chemicals used to evaluate the effectiveness of ASP formulations for producing Karamay reservoir oil. Concentrations are reported as active wt percent. Primary surfactant were selected based on results of phase behavior and IFT measurements in Phase 1. Cosurfactants were added, if required, to increase primary surfactant solubility.

Samples of Karamay formation brine (KFW), Karamay river water (KRW), and Karamay stock tank oil were provided by the Xinjiang Petroleum Administration (XPA). KFW and KRW were analyzed to determine composition and concentration of ionic components. The water compositions were then formulated to simulate these values. The composition of simulated KFW was altered slightly midway through the project to represent the measured ionic concentrations more closely. Table 2 shows the water formulations used for the Karamay ASP project.

Table 1 Oil Recovery Chemicals Used for the Karamay ASP Oil Recovery Project

Name	Chemical	Manufacturer	Activity, %	Additional Information
CJ–2 (primary surfactant)	Petroleum Sulfonate, Anionic	Xinjiang Petroleum Administration	45.2	Sulfonated crude oil
Petrostep B–100 (primary surfactant)	Alkyl aryl sodium sulfonate, Anionic	Stepan	56.3	MW ≈ 420 average R = C17
Neodol 25–12 (cosurfactant)	Alcohol Ethoxylate, Nonionic	Shell	100	12 EO's $-[C-O]_{12}-$ R = C12-C15
Neodol 25–3S (cosurfactant)	Alcohol Ethoxylated Sulfate, Anionic	Shell	58.75	3 EO's $-[C-O]_{3}-$ R = C12-C15
MO 3000H-SF	Polyacrylamide polymer	Mitsubishi	100	

Table 2 Water Analysis and Simulated Brine Formulations for the Karamay Oil Recovery Project

	Injection Water		Formation Brine		
	Karamay river water	Synthetic	Karamay formation brine	Synthetic #1	Synthetic #2
Calcium, mg/L	69	33	26	46	27
Magnesium, mg/L	10	10	38	32	27
Sodium, mg/L	40	44	2,420	2,320	2,322
Potassium, mg/L	2	25	20	30	30
Chloride, mg/L	23	87	2,130	1,460	2,191
Sulfate, mg/L	114	31	4	37	37
Carbonate, mg/L	—	6	—	93	—
Bicarbonate, mg/L	126	104	2,543	3,751	2,614
TDS, mg/L	337	340	6,146	7,768	7,283
pH	7.9	8.1	8.0	—	—

Formation Damage Study in Karamay Reservoir Rock

Karamay reservoir core S42-2 was initially saturated with 3% potassium chloride solution to minimize clay swelling and fines migration. Initial permeability was approximately 400 mD. However, pressure measurements never completely stabilized, and a very slow increase in pressure differential across the core was observed as the 3% KCl was pumped through the core. This suggests that some changes in the core were occurring even though a "nondamaging" brine was used.

After conducting a tracer test and injection of the formation brine, core permeability dropped to less than 200 mD. Permeability had declined during the tracer test where approximately 2 PV of formation brine had been used to push the tracer through the core. After an additional 1.6 PV of formation brine was injected, the permeability dropped to 188 mD. Figure 1 shows the change in core permeability with injection of formation brine.

Injecting Karamay river water caused a precipitous drop in permeability to brine. With less than 0.2 PV of river water injected, the permeability was less than 60 mD. Permeability continued to decline to approximately 25 mD after injection of 2.0 PV. This represents a permeability reduction of at least a factor of 10. Permeability was reduced 87% from that measured after injection of formation water and 94% of the initial value measured with 3% KCl. Permeability and pressure histories are shown in Figure 2.

KCl brine was reinjected to determine if permeability could be restored. The brine permeability

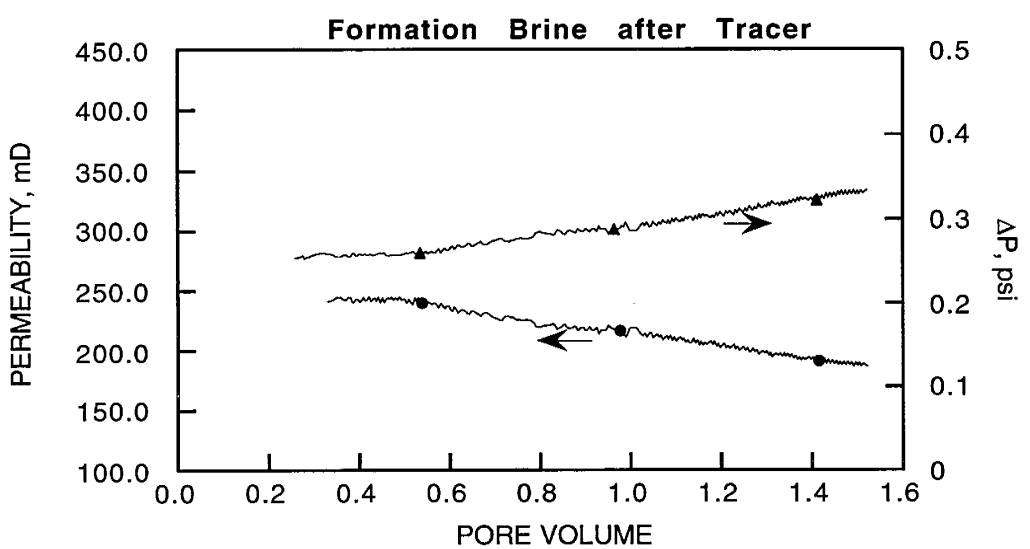

Figure 1 Change of Karamay Core Permeability with Injection of Simulated Formation Brine. Karamay reservoir core plug, S42-2, was used in this study.

did increase but not to the levels initially measured for the core. Permeability increased to approximately 51 mD and remained at that level as 2 PV of 3% KCl was pumped through the core. Subsequent injection of 2% NaCl, 5% KCl, and 3% NaCl caused minor fluctuations in permeability to brine without either major declines or major improvements in overall permeability. Most of the damage caused by the Karamay river water could not be reversed. Care should be taken in the field to prevent injection of low salinity water and subsequent damage to the reservoir.

A second Karamay reservoir core, Y-18-2, was saturated with 3% KCl and then contacted with 2% NaCl without first damaging the core with Karamay river water. Permeability to 3% KCl was 57 mD, and permeability was reduced by approximately 11% after injecting 1 PV 2% NaCl. Injecting 5 PV formation water further reduced brine permeability by a factor of 5. Core was then saturated with oil (k_{orw}= 83 mD) used for an oil recovery experiment rather than for further damage studies.

In summary, a series of brine solutions were pumped through a sample of reservoir rock from the Karamay reservoir. Permeability declined slowly as formation brine was pumped through the core. Introduction of Karamay river water caused a significant reduction in permeability which was only partially restored when the rock was contacted with a non damaging brine solution containing potassium chloride. Subsequent contact with 2% and 3% NaCl resulted in some reduction in permeability. These results indicate that problems can be anticipated if low salinity brine is introduced into the Karamay reservoir as part of an alkaline-surfactant-polymer project.

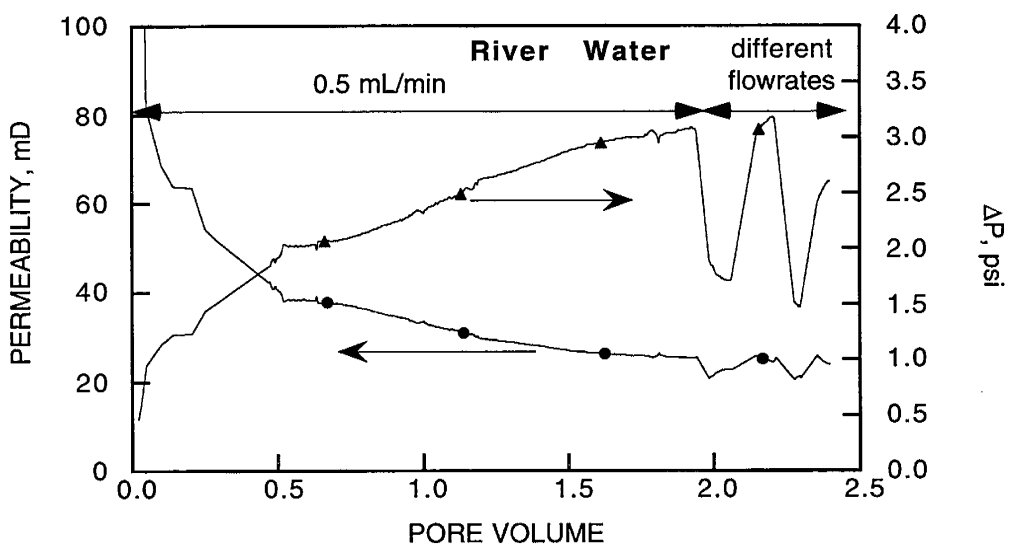

Figure 2 Pressure History and Permeability Change as a Function of Pore Volumes of Karamay River Water through the Core. Karamay reservoir eore plug, S42-2, was used in this study.

Oil Recovery in Berea Sandstone

Corefloods were conducted in 200 mD Berea sandstone to evaluate Karamay oil recovery potential using several different surfactant systems. Berea sandstone was used to provide a relatively uniform rock matrix for comparative studies of oil recovery for different chemical systems. For these tests, Karamay stock tank oil was diluted with n-hexane and iso-octane to reduce oil viscosity while maintaining the same oil-surfactant solvency as would occur with live crude. Oil recovery was measured for chemical formulations with and without surfactant and for chemical formulations at two levels of alkalinity. Table 3 summarizes the chemical systems used to recover Karamay oil from Berea sandstone. The most important results will be described in this section. Additional details of these experiments have been summarized in interim reports that are reproduced in Appendix B. Table 4 summarizes the Berea core properties used in this study, and Table 5 summarizes various oil saturations and recovery efficiencies obtained for the different tests.

Oil Recovery Using the B-100 Surfactant with 1.6% Na_2CO_3

Polymer (KCF-2) alone produced only 9% of the oil remaining after waterflood. Significantly greater recoveries were achieved when alkali or alkali and surfactant were used with the polymer. Figure 3 compares oil production for three chemical systems, polymer, alkaline-polymer (KCF-3), and surfactant-alkaline-polymer (KCF-1). Adding 0.5% (active weight %) B-100 surfactant to the alkaline-polymer slug (KCF-3) failed to improve oil recovery significantly. KCF-1 contained no polymer in the surfactant slug. An additional test (KCF-4) included polymer in the surfactant slug. Figure 4 compares oil production for these tests. In general, including polymer from the beginning of chemical injection appears to favor faster oil production but did not necessarily result in greater overall oil production. Total oil production during KCF-4 was slightly lower than the production during KCF-1, and the residual oil saturation after chemical flood was higher for KCF-4.

KCF-3, the chemical slug that contained only alkali and polymer performed as well or better than the systems that contained the B-100 surfactant. Possible adverse surfactant polymer interactions may affect the chemical system effectiveness.

Oil recovery efficiency (R_{eff}) was also evaluated in higher permeability Berea sandstone core. A large pore volume of the alkali-surfactant-polymer system containing B-100 surfactant followed by polymer was used to evaluate the maximum recovery expected for the most favorable recovery

Table 3 Chemical Floods Conducted in Berea Sandstone

Core I.D.	Brine, waterflood and post chemical flood	preflush [NaCl], %	Slug size PV	Chemical slug composition	Slug size PV	Mobility slug composition	Slug size PV
KCF-1	River water			0.5% B-100 1.6% Na2CO3	0.36	1500 ppm MO3000H-SF	1.10
KCF-2	Formation brine					1500 ppm MO3000H-SF	1.00
KCF-3	Formation brine			1.6% Na_2CO_3, P*	0.5	1500 ppm MO3000H-SF	0.74
KCF-4	Formation brine			0.5% B-100 1.6% Na_2CO_3, P	0.31	1500 ppm MO3000H-SF	1.00
KMAY-1	Formation brine			0.5% B-100 1.6% Na_2CO_3, P	0.69	1500 ppm MO3000H-SF	1.05
KMAY-2	Formation brine			5.0% CJ2 1.6% Na_2CO_3, P	0.31	1500 ppm MO3000H-SF	0.97
KMAY-4	Formation brine	1.5	0.2	1% CJ2 1.6% Na_2CO_3	0.44	1500 ppm MO3000H-SF 0.5% NaCl	1.20
KMAY-5	Formation brine	1.50	0.3	2% CJ2 1.6% Na_2CO_3, P	0.33	1500 ppm MO3000H-SF 0.5% NaCl	1.13
KMAY-6	Formation brine	3	0.21	2% CJ2 1.6% Na_2CO_3, P	0.31	1500 ppm MO3000H-SF	1.01
KMAY-7	Formation brine	1.5	0.21	2% CJ2 1.6% Na_2CO_3, P	0.31	1500 ppm MO3000H-SF 0.5% NaCl	1.05
radial core	Formation brine	3	0.3	2% CJ2 1.6% Na_2CO_3, P	0.32	1500 ppm MO3000H-SF followed by 1800 ppm MO3000H-SF	0.95
KCF-6	Formation brine	3	0.2	0.4% B-100 0.08% Neodol 25-3S, 2.5% Na2CO3, P	0.30	1500 ppm MO3000H-SF 1.0% NaCl	1.00
KMAY 3	Formation brine	3	0.26	0.45% CJ2, 0.068% Neodol 25-12, 2.5% Na_2CO_3, P	0.32	1500 ppm MO3000H-SF	0.98

*P = 1500 ppm MO3000H-SF

Table 4 Core Properties (Porosity and Permeability) for Experiments Conducted in Berea Sandstone Core

Core I.D.	Porosity, %	Initial k_b, mD	k_{orw}, mD	k_{wro}, mD
KCF-1	20.2	245	372	4.0
KCF-2	20.3	177	275	6.8
KCF-3	20.2	145	229	—
KCF-4	20.0	196	300	5.8
KMAY-1	23.2	416	668	32.8
KMAY-2	19.5	158	213	4.7
KMAY-4	19.6	207	291	—
KMAY-5	18.8	188	248	—
KMAY-6	19.6	165	230	5.6
KMAY-7	19.9	207	278	6.5
radial core	22.5	198	203	
KCF-6	19.9	100	96	4.6
KMAY 3	19.6	103	272	3.4

where k_b = brine permeability; k_{orw} = oil permeability at residual brine; k_{wro} = brine permeability at residual oil

Table 5 Oil Saturations and Recovery Efficiencies for Experiments Conducted in Berea Sandstone

Core I.D.	S_{orw}, %	S_{row}, %	S_{roc}, %	R_{eff}, % S_{row}	Flood type
KCF-1	66.6	31.7	15.2	52.1	ASP
KCF-2	64	36.7	32.5	9	A
KCF-3	65.4	34.5	15.8	54.2	AP
KCF-4	65.1	39.7	23.6	40.6	ASP
KMAY-1	66.3	31.2	10.2	67.3	ASP
KMAY-2	62.8	32.3	1.9	93.8	ASP
KMAY-4	68.8	41.4	11	74.2	ASP
KMAY-5	67.4	37.1	2.4	93.4	ASP
KMAY-6	67	43.2	9.2	78.8	ASP
KMAY-7	61.7	38.2	10.9	71.4	ASP
radial core	62.8	40.5	16.9	58.1	ASP
KCF-6	66.7	34.9	22.9	34.1	ASP- higher A
KMAY 3	68.7	41.1	24.9	39.3	ASP-higher A

where S_{orw} = oil saturation at residual water; S_{row} = oil saturation after waterflood; S_{roc} = oil saturation after chemical flood; and R_{eff} = recovery efficiency

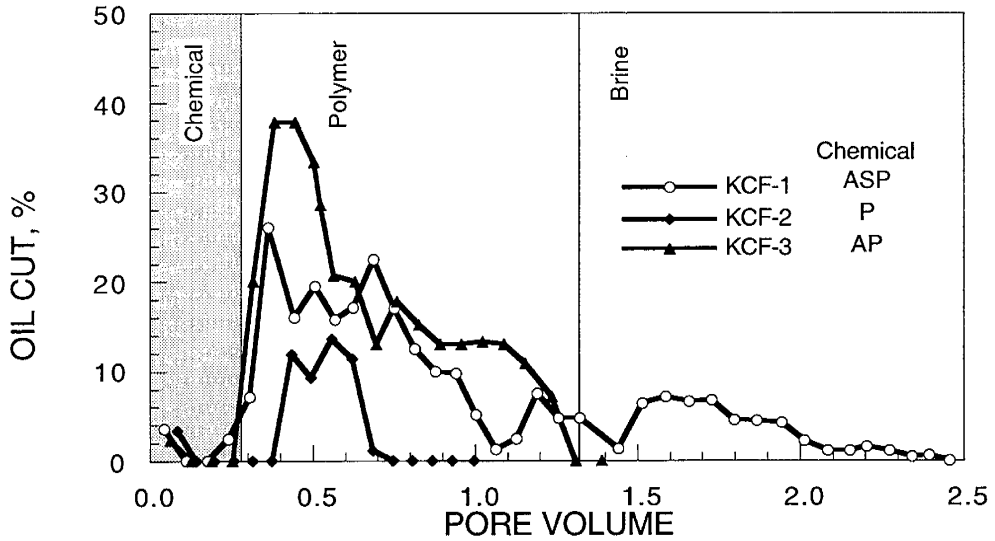

Figure 3 Comparison of Karamay Oil Production from Berea Sandstone Core for Polymer, Alkaline-Polymer, and Alkaline-Surfactant-Polymer Chemical Slugs.

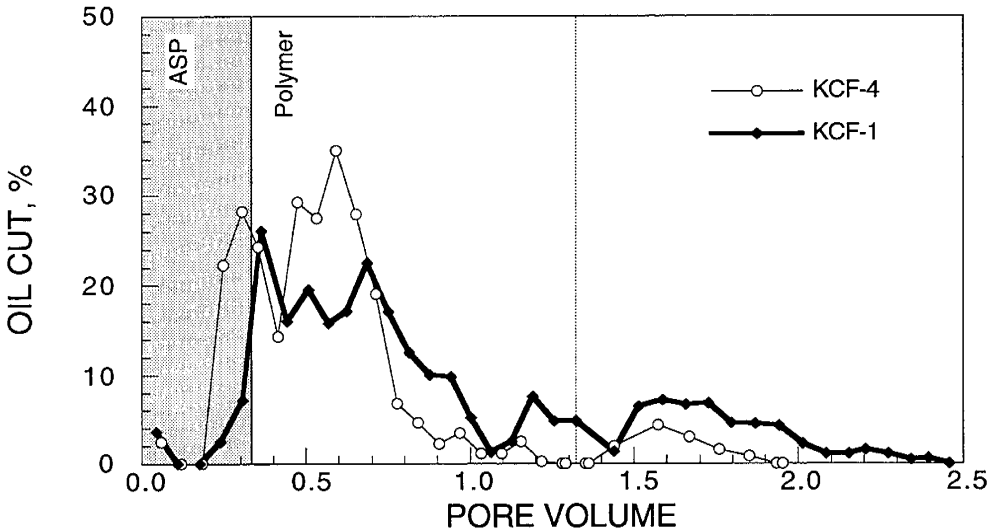

Figure 4 Comparison of Karamay Oil Production from Berea Sandstone Core for Surfactant Slugs with Polymer (KCF-4) and without Polymer (KCF-1).

conditions. Oil recovery for this test, KMAY-1, was greater than the tests conducted in lower permeability Berea sandstone. Use of an excessive amount of chemical, however, did not recover all the oil from the core. Figure 5 shows the oil recovery for this test. R_{eff} was 67% as compared to 41% for the test in lower permeability core (KCF-4).

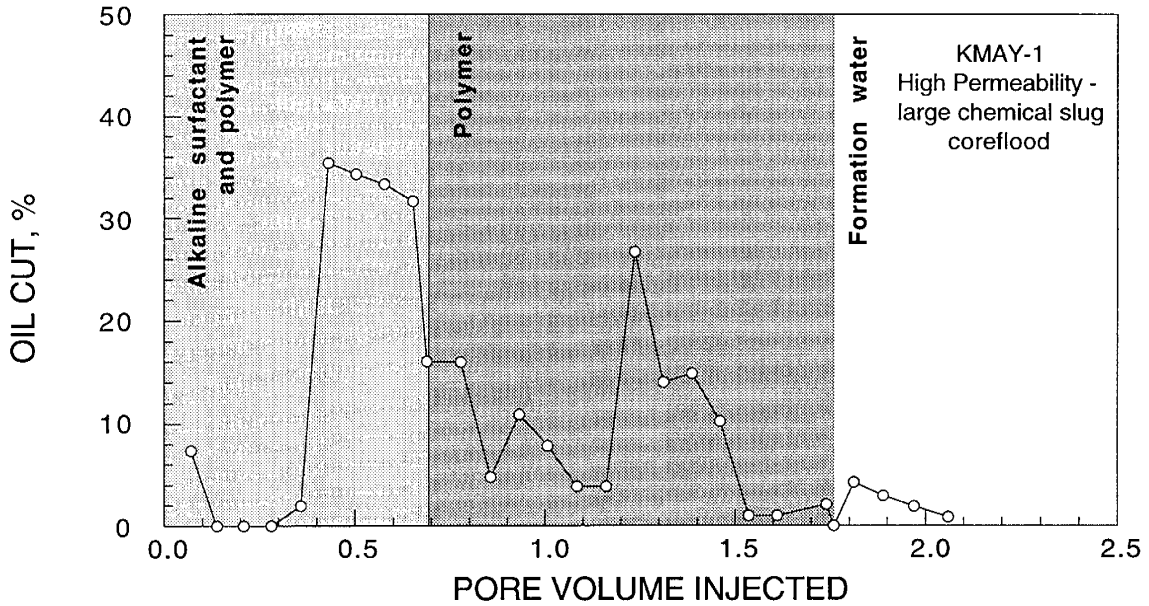

Figure 5 Oil Cut as a Function of Fluid Injected for Production of Karamay Oil in a High Permeability Berea Sandstone Core Using a Large Chemical Slug. Chemical Formulation is the ASP Using 0.5% B-100 and 1.6% Na_2CO_3.

Oil Recovery Using B-100 at Higher Alkalinity Concentration

Identification of several problems during this project suggested that the oil recovery potential of ASP systems designed for higher ionic strengths and sodium carbonate concentrations. These problems included possible formation damage from injection of slugs made with low salinity Karamay river water and possible consumption of alkalinity in the reservoir. Increasing the ionic strength and/or carbonate concentration affects surfactant solubility and phase behavior characteristics, however. Addition of small amounts of ethoxylated surfactants to the primary surfactant system can result in a surfactant system suitable for the increased alkalinity of the chemical slug.

Ratios of B-100 and an ethoxylated anionic surfactant, 0.08% Neodol 25-3S, were prepared and screened with Karamay oil and sodium carbonate to determine the most favorable regions of interfacial tension. Figure 6 shows IFTs for different B-100/Neodol 25-3S surfactant mixtures.

An ASP formulation that included 2.6% Na_2CO_3, 0.4% B-100, 0.08% Neodol 25-3S, and 1,500 ppm MO3000H-SF polymer was found to maintain a very low IFT, 0.3 μN/m. The IFT remained low when the formulation was diluted with formation brine. A coreflood (KCF-6) was conducted in Berea sandstone to evaluate the chemical effectiveness and compare with previous coreflood conducted at lower salinity. The injection strategy included injecting a 3% NaCl preflush

and adding 1% NaCl in the mobility control polymer after the ASP slug to provide a salinity gradient during chemical injection. Oil recovery, however, was no better than a similar test conducted at lower salinity. Figure 7 shows the oil cut for this oil recovery experiment. Oil recovery efficiency was 34% which was less than the 41%–52% obtained for 0.5% B-100 using 1.6% Na_2CO_3.

Chemical System Containing CJ2 Surfactant

Surfactant manufactured by the XPA was also supplied to determine suitability for recovery of Karamay oil. IFT measurements during Phase 1 indicated that this surfactant did not generate IFT values as low as those generated by the B-100 surfactant. An additional sample of the surfactant was received in September 1994, and oil recovery experiments were conducted using the new CJ2 sample.

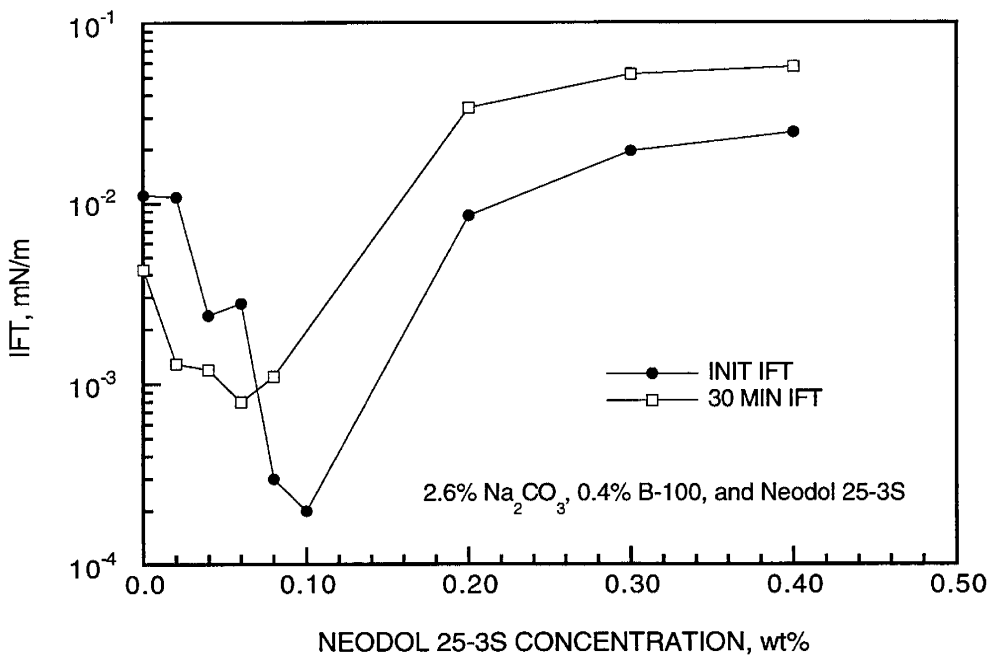

Figure 6 IFT as a Function of Neodol 25-3S Surfactant for the Chemical System, 2.6% Na_2CO_3 and 0.4 B-100 in Karamay River Water.

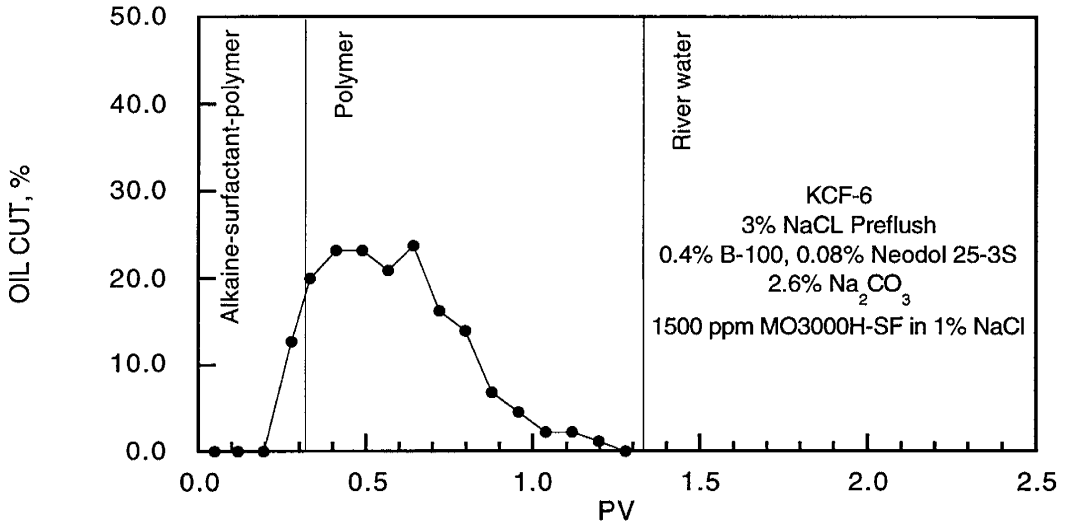

Figure 7 Oil Cut as a Function of Pore Volume of Injected Fluid for the Chemical System Designed for Higher Salinity. A 0.2 PV slug of 3% NaCl was injected in front of the chemical slug.

The initial coreflood in Berea sandstone using the CJ2 surfactant was designated KMAY-2. The chemical system was designed for the lower alkalinity concentration of 1.6% Na_2CO_3 and a surfactant concentration level of 0.5% CJ2. A higher surfactant concentration (5.0%) was actually used for the test. Oil recovery was very high, and production exceeded that from previous tests using B-100 surfactant. Figure 8 shows oil production as a function of the amount of fluid injected. The recovery efficiency was 94% of *Srow*. Final oil saturation was very low (*Sroc* = 1.8%) at the completion of the test. This saturation level was significantly lower than the coreflood using B-100 surfactant (*Sroc* = 15.2%). For CJ2, oil recovery was aided by injecting excess surfactant.

Two problems were evident using the high concentration of CJ2. Surfactant-polymer interactions were much more evident in the chemical slug. The chemical system had to be stirred continuously during injection since components would partially separate or precipitate fairly rapidly. Figure 9 shows a picture of the ASP solutions prepared with different surfactant concentrations. At a very low surfactant concentration, phase separation occurred. As surfactant concentration increased, material precipitated. Analysis of the precipitated material indicated the presence of most of the surfactant and some of the polymer. Surfactant-polymer interactions could result in injecting an unknown amount of chemical, altering solution properties such as viscosity, injection problems, especially into lower permeability rock.

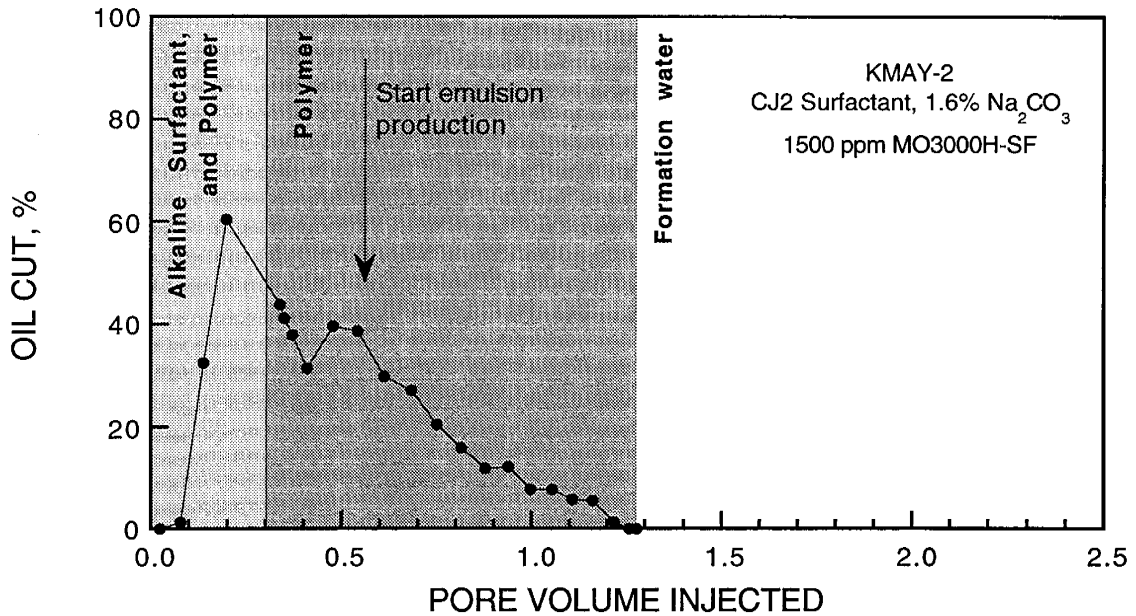

Figure 8 Oil Cut as a Function of Fluid Injected for Production of Karamay Oil Using a Chemical Slug that Contained 1.6% Na_2CO_3, 5.0% CJ2 Surfactant, and 1500 ppm Polyacrylamide, MO3000H-SF. Note the oil cut scale is greater than that for the previous graphs.

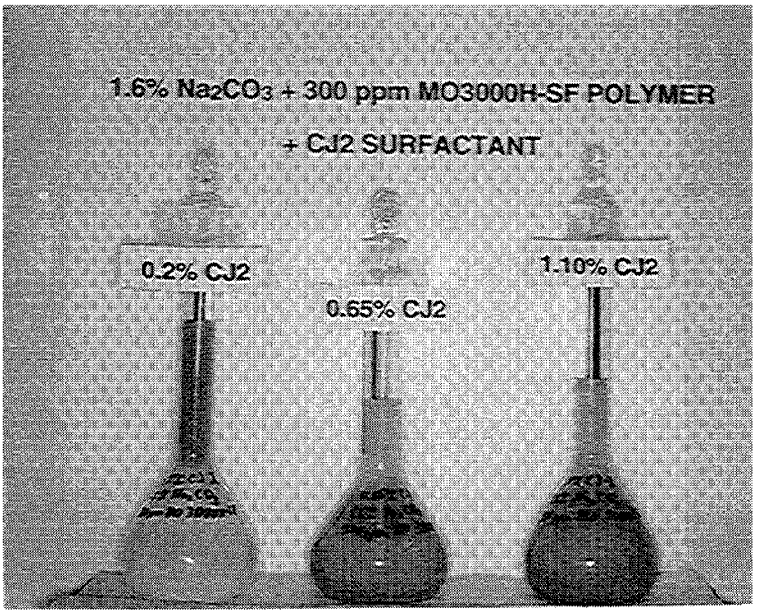

Figure 9 Surfactant-Polymer Interactions in ASP Solutions as a Function of Surfactant Concentration. Initially, Material is Lower Density than the Solution. As surfactant doncentration increases, the material precipitates.

The second problem observed with higher concentration surfactant was the production of emulsions. As noted in Figure 8, after the initial oil bank, oil was produced as an emulsion that did not spontaneously separate. Alcohol (2-propanol) was added to the emulsion, and each fraction was centrifuged to separate the oil and water. Using excess surfactant in the chemical slug resulted in producing excess surfactant, which was then available for producing water and oil emulsion. If excess surfactant was produced in a field application, evaluation of emulsion breaking chemicals would be required.

A series of experiments was then conducted to determine the minimum amount of CJ2 that would be required to maximize Karamay oil production from Berea sandstone core. Corefloods KMAY-4 and KMAY-5 were conducted using 1% and 2% CJ2 surfactant with alkali and polymer. As surfactant concentration decreased, oil recovery efficiency decreased and oil saturation at the end of the chemical flood increased. (The summary of oil saturations is shown in Table 5.)

Results of the tests are summarized in Figures 10 and 11. The open circles represent tests conducted using 0% surfactant, 0.5% B-100 surfactant, and 1, 2 or 5% CJ2 surfactant in 1.6% Na_2CO_3 and 1500 ppm polymer. The tests were conducted in core with initial permeability of approximately 180 mD or more. Oil recovery increased (residual oil saturation decreased) significantly as the surfactant concentration in the chemical slug was increased from 0.5% to 2% by weight. A further increase to 5% did not appreciably increase oil recovery. In addition, using a surfactant concentration of 5%, a significant amount of oil was produced as an emulsion. For lower concentration surfactant, most of the oil was produced before the emulsion was observed.

Several additional tests have been conducted using the 2% CJ2 system. In both cases, KMAY-6 and KMAY-7, oil saturation after the chemical flood and recovery efficiency were not as favorable as the results from KMAY-5. Residual oil saturation after chemical flood was 9.2% for KMAY-6 and 10.9% for KMAY-7 rather than 2.4% for KMAY-5. Recovery efficiency was 78.8% for KMAY-6 and 71.4% for KMAY-7 rather than the 93.4% for KMAY-5. Figure 12 compares oil cuts for the three tests.

Several factors may have contributed to variations in recovery success. Mobility control differed for the three tests. Better mobility control was provided for KMAY-5 than for KMAY-6 or KMAY-7. Mobility calculation are discussed in Appendix 5. Another potentially serious problem was the surfactant-polymer interaction that caused chemical components to separate before injection into the core. Failure to maintain a uniform chemical slug resulted in injecting varying amounts of chemical. The chemical slug for KMAY-5 was stirred continuously during injection. The chemical

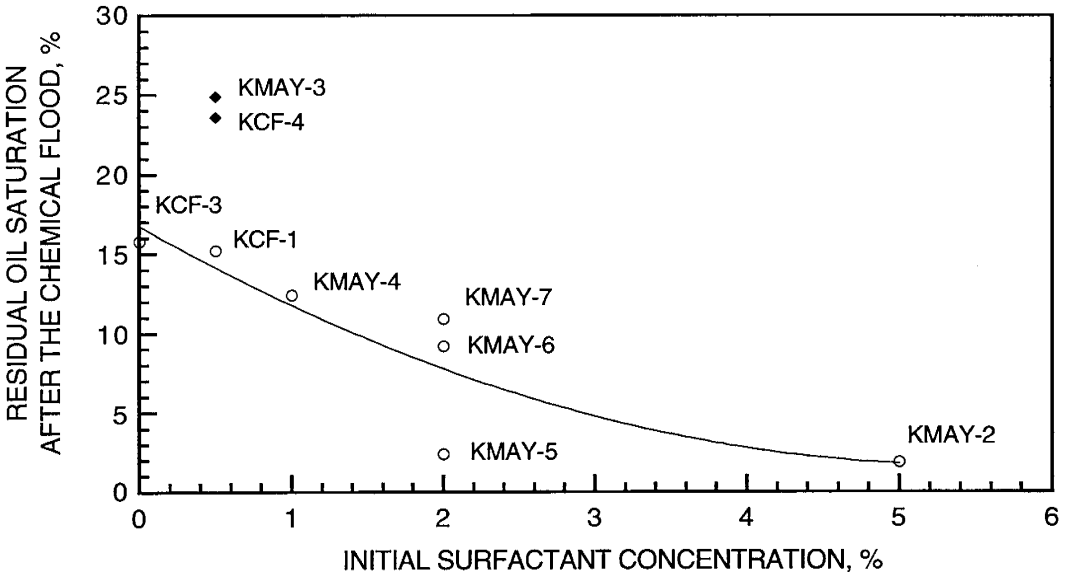

Figure 10 Residual Oil Saturation after Chemical Flood as a Function of CJ2 Surfactant Concentration.

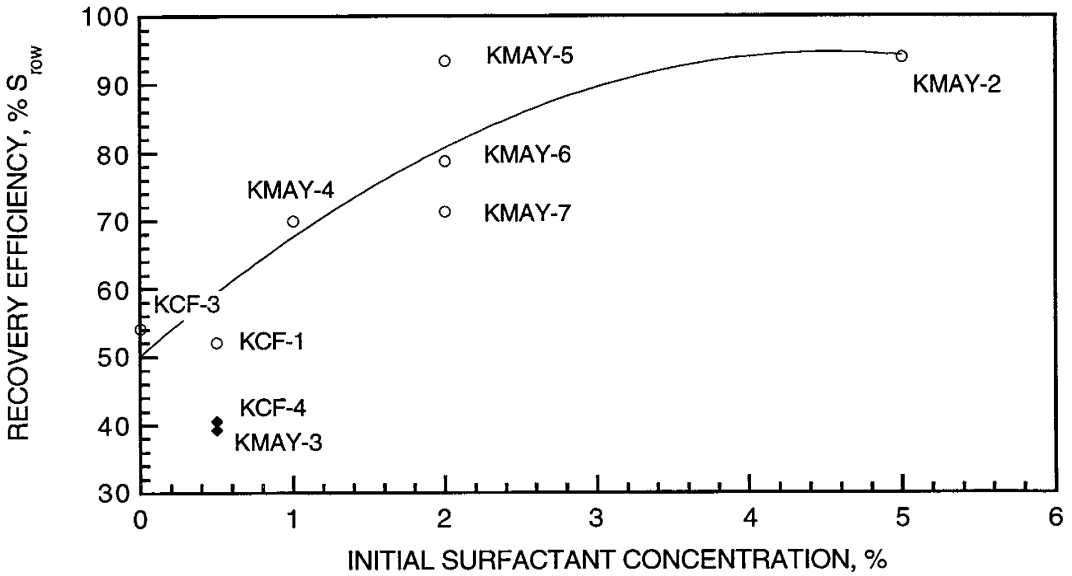

Figure 11 Oil Recovery Efficiency as Percent of Oil Remaining after the Waterflood as a Function of CJ2 Surfactant Concentration.

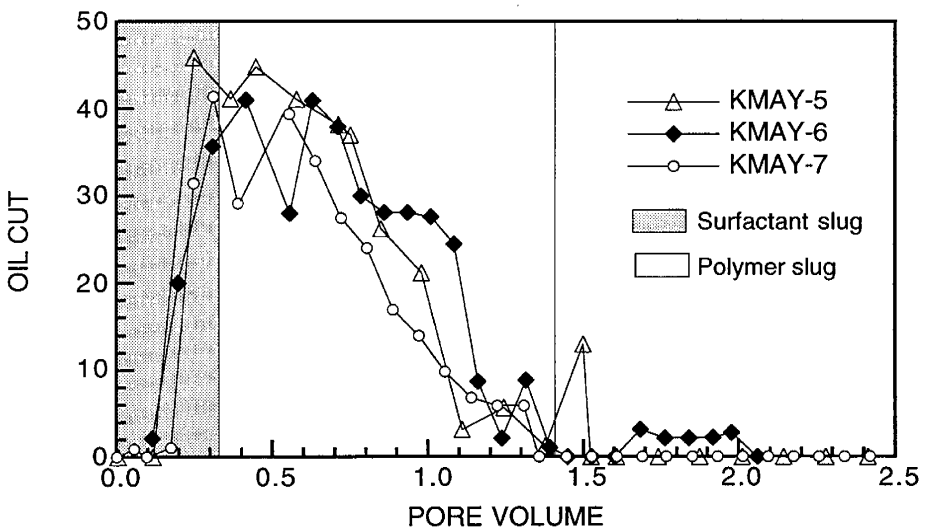

Figure 12 Comparison of Karamay Oil Production for 3 tests Using the Chemical System 2% CJ2 Surfactant, 1.6% Na_2CO_3 and 1,500 ppm Polymer.

slugs for KMAY-6 and KMAY-7 were agitated but not stirred continuously. The surfactant-polymer interaction could cause problems injecting chemical in the field. From the shape of the production curve, a less effective polymer mobility slug may have occurred with KMAY-7 since production tapered off earlier for KMAY-7 than for the other tests.

Karamay oil recovery was also evaluated in a 200 mD radial Berea core using the 2% CJ2-1.6% Na_2CO_3-1,500 ppm polymer chemical system. The chemical slug was initially followed by 1,500 ppm polymer. Concentration of the polymer was then increased to 1,800 ppm. Recovery efficiency was only 58% rather than 71%–93%, and oil saturation after the chemical flood was 16.9% rather than 2.4%–10.9%. An increase in polymer concentration failed to produce more oil from the radial core. Breakthrough in one flow direction may occur earlier than in other directions, reducing overall sweep efficiency of the fluids. The same batch of chemicals was used in the KMAY-7 linear Berea coreflood experiment. Figure 13 shows a comparison of oil production for the radial versus the linear coreflood. Oil production was actually very similar for the two tests.

CJ2 Surfactant and Higher Alkalinity System

A coreflood, KMAY-3, was conducted in Berea sandstone core using the a CJ2 surfactant system designed for use with higher alkalinity concentrations. As with the B-100 surfactant system designed for higher alkalinity, the reasons for investigating a change in chemical slug composition was to increase the salinity of injected fluids to reduce possible formation damage from clay problems and to inject additional alkalinity to offset chemical consumption in the reservoir. Because of the higher ionic strength of the solutions, a second surfactant was added to the chemical

formulation to increase the solubility of the CJ2 surfactant. The surfactant mixture used in this test was 0.45% CJ2 and 0.068% Neodol 25-12, a nonionic ethoxylated surfactant. Figure 14 shows the time dependent IFT of this surfactant mixture in 2.5% Na_2CO_3.

The injection strategy included injecting 3% NaCl preflush to increase salinity, followed by the mixed surfactant in 2.5% Na_2CO_3 and polymer. The polymer mobility control slug contained 1% NaCl. Recovery efficiency was 39%, and the final oil saturation after chemical flood was 25%. Recovery compared with that obtained by the chemical system using B-100 surfactant at higher salinity (KCF-6). Figure 15 shows a comparison of the oil production data for KMAY-3 and KCF-6. Oil production in Berea sandstone using the higher salinity surfactant slugs was, in most cases, not as high as production using the lower alkalinity surfactant slugs. Attempts to protect the reservoir by increasing salinity of injected chemicals or attempts to improve surfactant solubility by adding cosurfactants would require further study to improve oil recovery potential of higher salinity chemical systems.

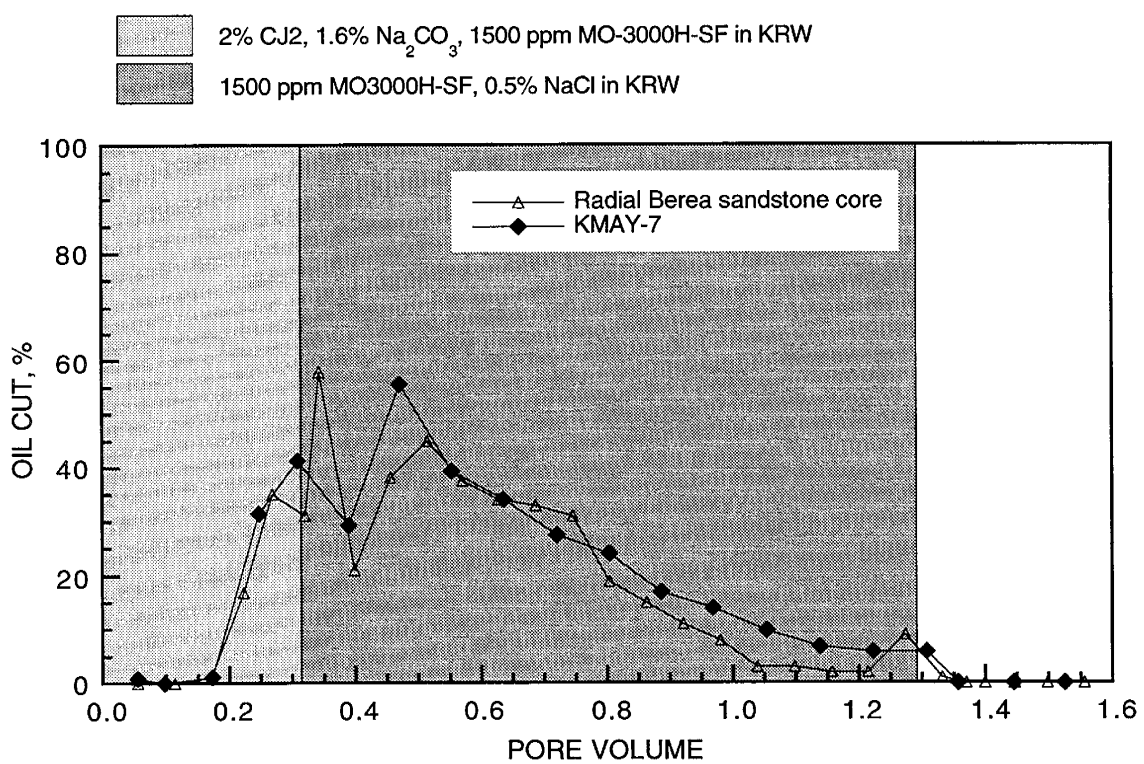

Figure 13 Comparison of Oil Cuts from the Radial Coreflood and Linear Coreflood Conducted in Berea Sandstone Using the Same Chemicals.

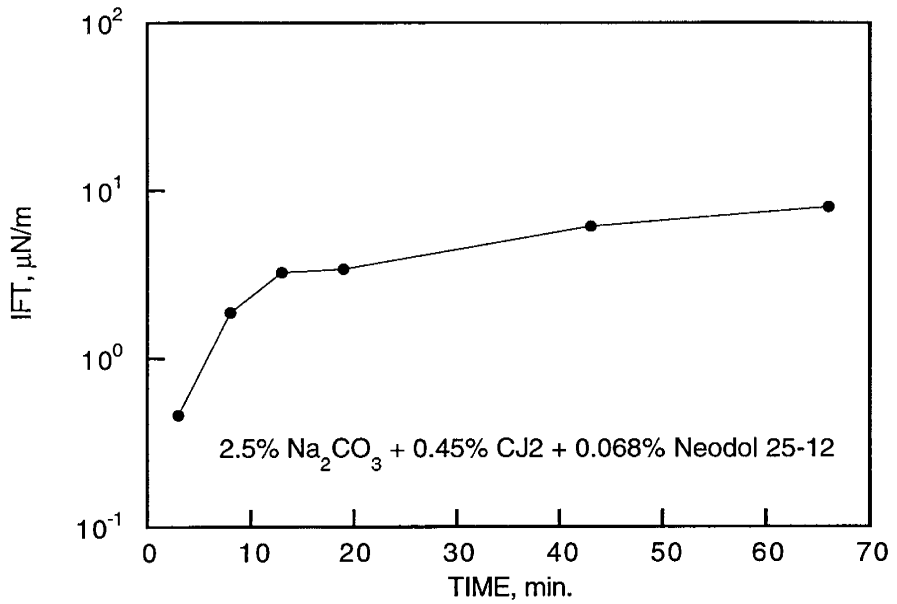

Figure 14 Time Dependent IFT for the CJ2 and Neodol 25-12 Mixture with Diluted Karamay Crude Oil.

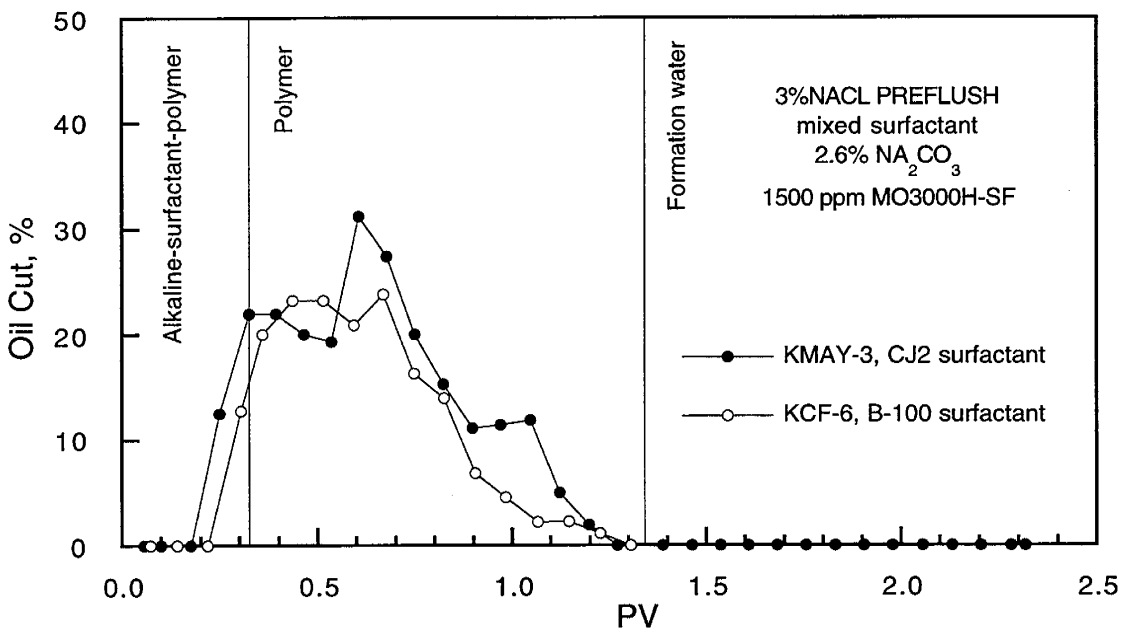

Figure 15 Comparison of Oil Production for ASP Karamay Oil Recovery Experiments Using 2.5% Na_2CO_3. Polymer for KCF-6 contained 0.5% NaCl and for KMAY-3 contained no added NaCl. Calculated mobility control differed little for these two tests, however. (See Appendix 5)

Oil Recovery in Karamay Reservoir Core

Core plugs 3.8 cm in diameter and approximately 5–8 cm long were received from XPA for use in the Karamay ASP evaluation project. A core identification list is reproduced in Appendix 1. Core quality was relatively poor. A number of pieces contained large pebbles. Many core plugs were of low permeability. A number of other cores were friable and disintegrated on handling. Poor quality core increased the time required to obtain oil recovery results using reservoir core and affected interpretation of oil recovery results.

Consolidated Karamay Reservoir Rock

Several oil recovery experiments were conducted using Karamay reservoir core. Difficulties were experienced achieving high oil saturations in the core plugs. Several methods were tried to increase oil saturations, including injecting higher viscosity, undiluted Karamay reservoir stock tank oil, followed by reduced viscosity Karamay oil, and using of a capillary plate to help remove water from the core. Subsequent measurement of core compressibility under laboratory conditions as described in Appendix 2 indicated that the pore volumes obtained by weight measurement may be slightly high by 5%–10% relative to pore volume under confining stress. This would increase calculated oil saturations slightly.

Additional problems were experienced handling friable core and selecting high-permeability core. To obtain additional oil recovery information, therefore, two tests were conducted in sandpacks prepared from crushed reservoir rock.

Finally, a coreflood was conducted using three core plugs stacked in an aluminum Hassler-sleeve coreholder so that oil saturations could be monitored during the course of chemical injection using CT-imaging techniques.

Table 6 summarizes chemical formulations, oil saturations, and recovery efficiencies for all tests conducted in Karamay reservoir core.

An AP chemical system containing 1.6% Na_2CO_3 performed poorly in a single core test, Y18-2, producing only 14% of the oil remaining after waterflood. This is considerably less than the AP system produced from Berea sandstone (54% R_{eff}).

Surfactant systems designed for the higher alkalinity conditions, 2.5% Na_2CO_3, produced approximately the same or slightly better than the same systems in Berea sandstone core. A comparison of recovery efficiencies is shown in Table 7.

Table 8 Results of Karamay Oil Recovery Tests Using Karamay Reservoir Core

Karamay core	Initial S_{or}, %	S_{row}, %	S_{roc}, %	R_{eff}, %	Surfactant, wt% *	Na_2CO_3, wt%
S42-2	54	26	14	42	0.45 CJ2 0.068 Neodol 25-12	2.5
Y2-1	90	32	18	46	0.4% B-100 0.08% Neodol 25-3S	2.5
Y18-2	59	24	21	12	none	1.6
Y1-1	56	15	6.2	60	2% CJ2 polymer not in surfactant slug	1.6
Stacked core	48	23	20	14	2% CJ2**	1.6
Sandpack #1	63	21	6.8	68	2% CJ2	1.6
Sandpack #2	52	15	3.6	76	2% CJ2 polymer not in surfactant slug	1.6

where S_{orw} = oil saturation at residual water; S_{row} = oil saturation after waterflood; S_{roc} = oil saturation after chemical flood; and R_{eff} = recovery efficiency

* Unless noted surfactant slug contained 1,500 ppm polyacrylamide (MO3000H-SF) along with the alkali. A mobility buffer of 1,500 ppm polymer followed the AP, AS, or ASP slug.

** Surfactant-polymer interaction resulted in injection of unknown amount of surfactant during test

Table 7 Oil Recovery Efficiencies in Berea Sandstone and Karamay Reservoir Rock Using the 2.5% Na_2CO_3 ASP Chemical Systems

Coreflood	Surfactant	R_{eff}, % S_{row}
KMAY-3 Berea sandstone	0.45% CJ2, 0.068% Neodol 25-12	39.3
KCF-6 Berea sandstone	0.4% B-100, 0.08% Neodol 25-3S	34.1
S42-2, reservoir core	0.45% CJ2, 0.068% Neodol 25-12	42
Y2-1, reservoir core	0.4% B-100, 0.08% Neodol 25-3S	46

The core Y2-1 was saturated with oil without cleaning and without first saturating with brine in order to obtain a high initial oil saturation. After completion of the test, the core was cleaned to obtain the initial pore volume. As a result oil saturation at the end of the waterflood was close to that obtained for many of the Berea coreflood tests (32%). The chemical system then reduced oil saturation to 18%.

A final single plug coreflood test was conducted with a reasonably uniform core, Y1-1, with initial permeability to brine of 115 mD. Recovery using the chemical system, 2% CJ2-1.6% Na_2CO_3 without polymer in the surfactant slug followed by 1,500 ppm polymer, achieved the highest recovery efficiency for any of the single core experiments. Very little oil was left in the core. This compares with the results from Berea corefloods, KMAY-4, -5, and -6 that used the same chemical system to produce Karamay oil. No surfactant, however, was detected in the core effluent, suggesting excessively high surfactant losses.

Sandpack Corefloods

Two Karamay oil recovery tests have been conducted in sandpacks using the chemical system of 2% CJ2 surfactant, 1.6% Na_2CO_3, and 1,500 ppm MO3000H-SF. The sandpacks, 1.9 cm in diameter and 21 cm long, were packed with crushed Karamay reservoir core that had been sieved to remove large (>18 mesh) pebbles and very fine (<200 mesh) material. Care was taken to minimize grinding the easily disaggregated core material to prevent excessive exposure of clays and loss of very fine material. Large rock fragments and pebbles were very hard and were difficult to grind to pass the 18 mesh sieve. This fraction of the core material was not included in the crushed core used in the sandpacks. The 18-200 mesh material from four cores was combined to provide crushed core for several coreflood tests. For the combined samples, the percentage of material by weight in each size range was 36.6%, >18 mesh; 60.3%, 18–200 mesh; and 3.1%, < 200 mesh.

In sandpack test #1 polymer was included in the surfactant slug and in sandpack test #2 no polymer was included in the surfactant slug. For test #1, oil saturation at the end of the oilflood was approximately 60%. For test #2, however, an oil saturation higher than 50% was not obtained. The oil saturation at the end of the waterflood was higher for sandpack test #1 than for sandpack test #2. Recovery efficiencies for the two tests were similar. Final oil saturation at the end of the chemical flood was slightly lower for test #2 than for test #1. Table 8 summaries oil saturations at various stages during the two sandpack tests.

Table 8 Summary of Oil Recovery Coreflood Data for Crushed Karamay Core Sandpack Tests

Sandpack Test	k_b, D	Oil saturation at the end of the oilflood, S_{orw} %	Oil saturation at the end of the waterflood, $Srow$ %	Oil saturation at the end of the chemical flood, $Sroc$ %	Recovery efficiency, R_{eff}, %
#1	2.5	62.8	21.2	6.8	68.2
#2	2.6	51.5	15.0	3.6	75.8

Nonuniform oil saturation distributions may account for the differences in oil saturations after oilflood and waterflood for the two tests. Figure 16 compares the oil cuts during the chemical flood for the two tests. The initial oil production cut for test #1 were significantly larger than those for test #2, possibly a result of the higher oil saturation at the end of the waterflood for test #1. Otherwise oil production is very similar for the two tests. S_{roc} compares with the previously reported values obtained for this chemical system in consolidated Berea sandstone core.

Stacked Core Coreflood

A Karamay oil recovery experiment was conducted in reservoir core using CT scanning to monitor oil movement during chemical injection. Three pieces of Karamay reservoir core were placed in an aluminum Hassler-sleeve coreholder with some crushed core added between core plugs to provide continuity from core to core. Fill material (18 to 200 mesh) was required because it was difficult to cut flat ends on the core plugs. The crushed core was used to fill the gaps caused by the uneven core ends. Difficulties were also experienced loading the cores. One piece that was originally selected for this experiment disintegrated during the loading process. The core selected to replace this piece was less permeable. The core had improved handling characteristics but may have adversely affected chemical injectivity and oil production.

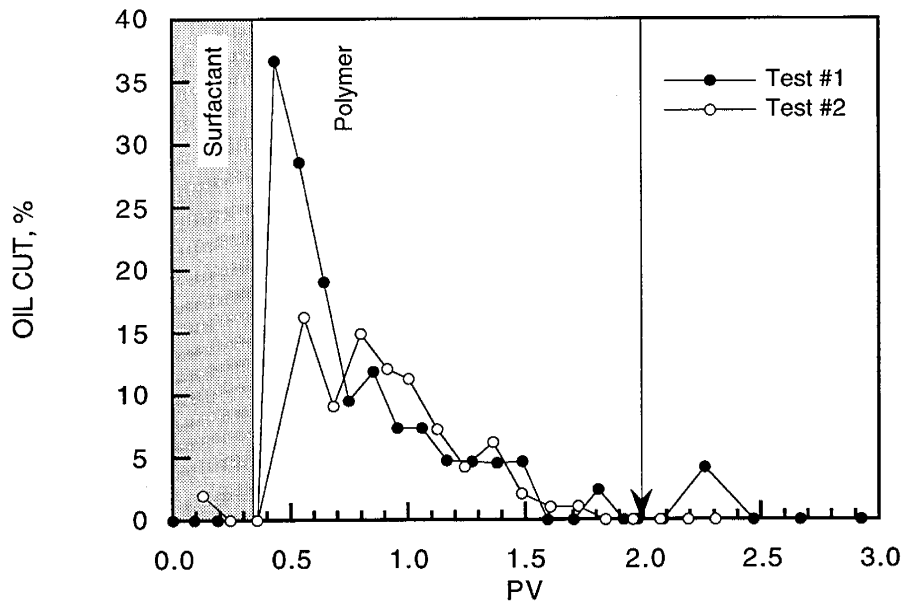

Figure 16 Comparison of Oil Cuts for Karamay Crushed Core Chemical Flooding Experiments.

Initial permeability to 3% KCl brine for the stacked cores was 57 mD. Permeability to oil at residual water saturation was 7.3 mD. After waterflood, permeability to formation brine was approximately 0.8 mD. The low permeability of the stacked core caused chemical injectivity problems.

Figure 17 shows the average porosity for the Karamay stacked core, and the porosity distribution along one vertical core slice. Areas of low porosity were seen in the regions where large pebbles were found. The areas between the core show as high porosity regions. A higher porosity region also exists in the most uniform core, Y2-2.

During chemical slug injection, pressure increased significantly. Fluid injection rates had to be continuously lowered to control the rising pressure. Average injection rate was less than 0.2 ft/D. Chemical slug injection, therefore, was extremely slow. This allowed extra time for the adverse surfactant-polymer interactions to form insoluble materials that separated from solution. As a result, a significant fraction of the injected surfactant slug contained lower than designed surfactant concentration and solution viscosity.

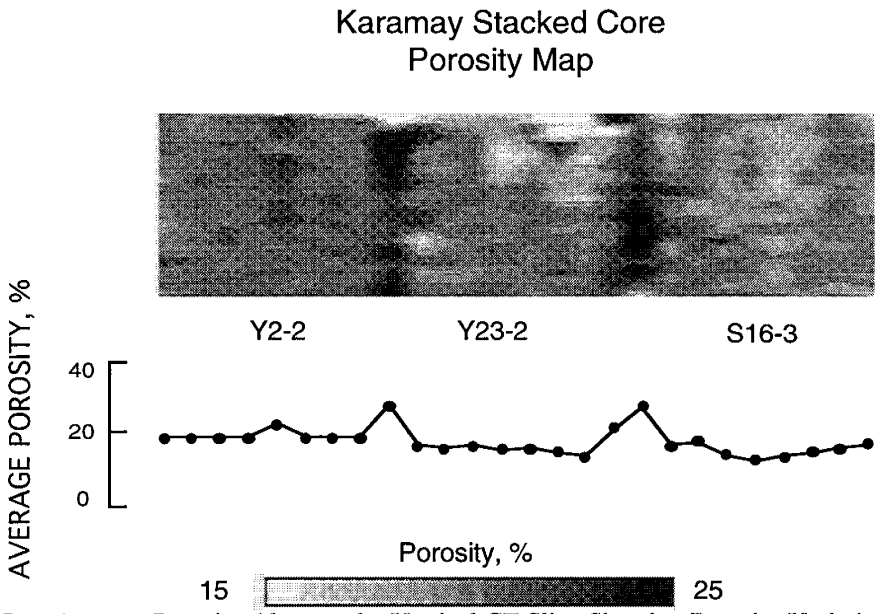

Figure 17 Average Porosity Along and a Vertical CT Slice Showing Porosity Variations in Stacked Karamay Reservoir Core for ASP Experiment.

Oil production after chemical flood was approximately 14% of the oil remaining after waterflood. The CT images of oil saturation distribution indicated some oil was mobilized. Oil saturation in the first core plug was reduced. Oil could be observed moving through the middle core. It appeared, however, that oil moved through the third core with difficulty. Oil saturation in the crushed core between cores 2 and 3 remained high throughout the test. Insufficient chemical injection or low core permeability may have caused the low production results.

The coreflood information is summarized in the attached stacked core coreflood report. Oil recovery was very poor. Figure 18 shows the oil cut as a function of injected fluid. Oil recovery efficiency at the end of fluid injection was approximately 14% of residual oil saturation.

CT determinations of oil saturations during the test indicated that the oil saturation distribution after the waterflood was very heterogeneous. Figure 19 shows average saturations along the core for various stages of the coreflood. Oil saturation between the core plugs remained high (average > 45%). At the end of the surfactant injection (0.5 PV), some oil had moved from the upstream core, Y2-2, to the space between cores. A small amount has been produced from core S16-3. After injecting 0.7 PV polymer, average oil saturations in the cores reduced slightly, but oil saturation in between cores was hardly affected. Oil in the higher porosity area of the upstream core, Y2-2, also did not appear to mobilize.

For a number of reasons, including low core permeability, poor fluid compatibility, poor injectivity, and injecting less than the desired amount of chemicals, the oil recovery results from this coreflood were discouraging. Simulation of this experiment would probably provide little useful information.

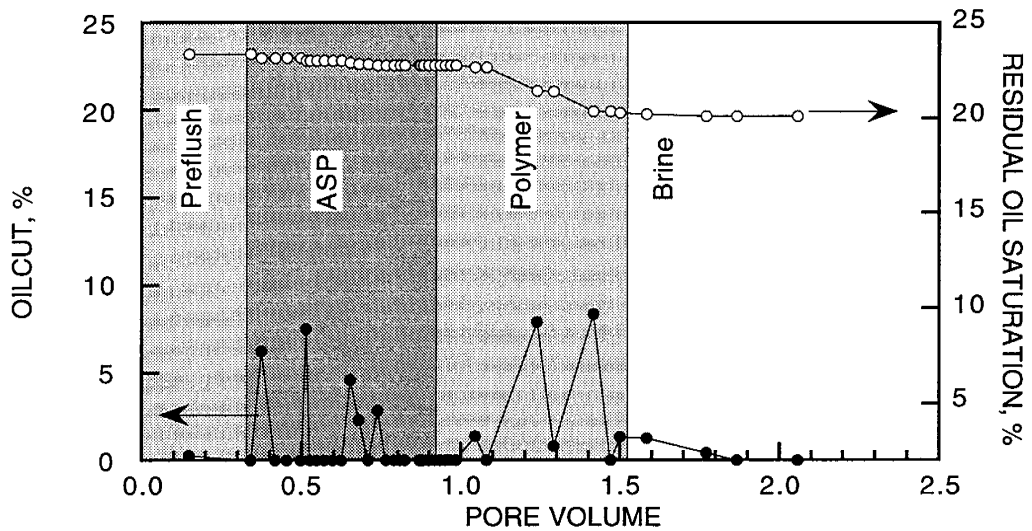

Figure 18 Oil Cut and Residual Oil Saturation During Karamay Stacked Core ASP Experiment.

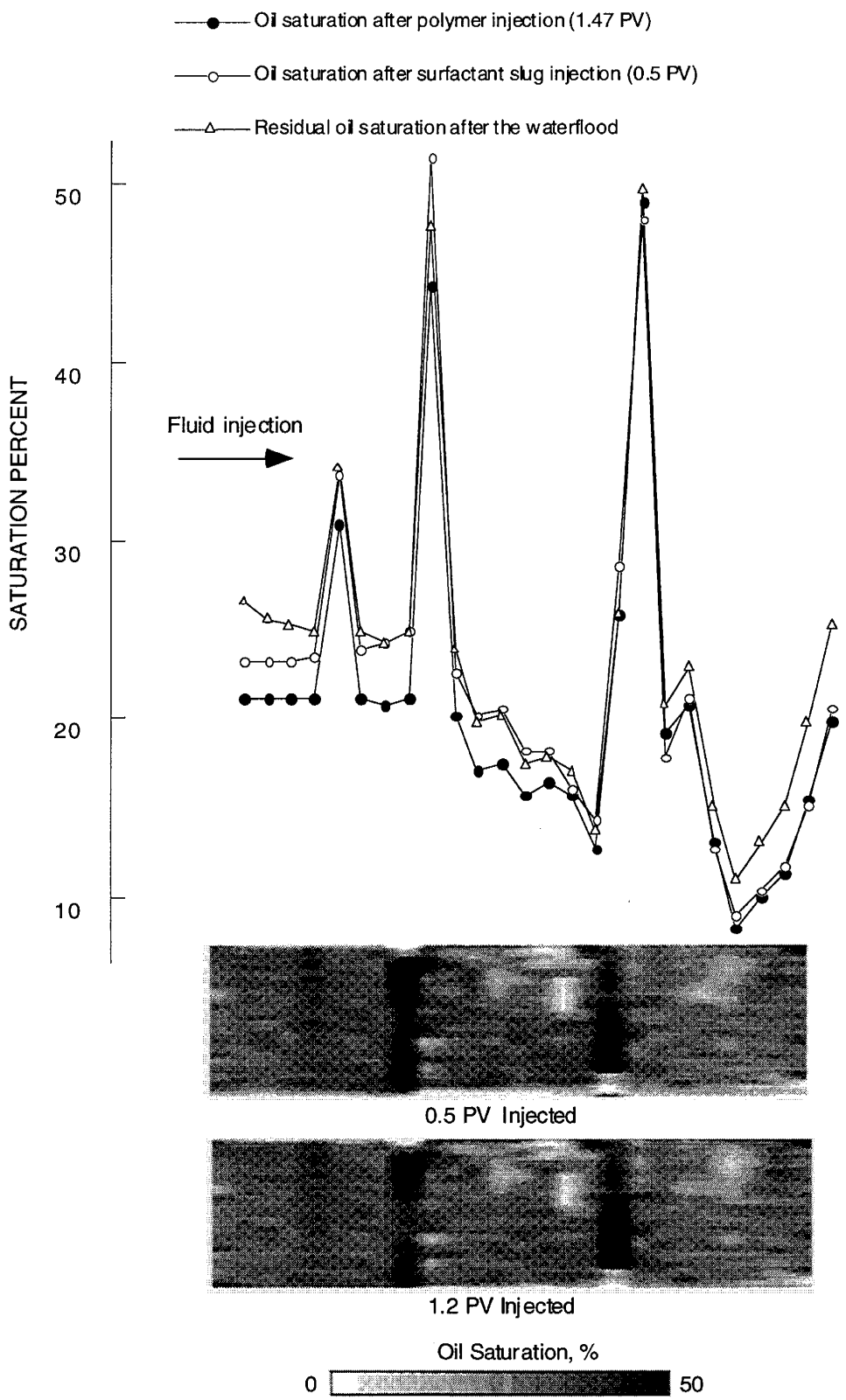

Figure 19 Average Oil Saturations Along the Stacked Karamay Core for Different Stages of an ASP Experiment. Oil Saturation Distributions in One Vertical Plane are Shown.

Chemical Adsorption Studies

Both static and dynamic adsorption measurements of alkalinity consumption, surfactant adsorption, and polymer adsorption were conducted as part of the Karamay ASP feasibility study.

Alkali Consumption

Alkali consumption measurements were determined for samples prepared by contacting crushed samples of Karamay reservoir rock with 0.01N Na_2CO_3 (approx. 0.053% Na_2CO_3). The crushed material was a mixture from core samples labeled as Y16-3, Y20-2, and Y20-1. Large pebbles of core were excluded from the sample of crushed material. The excluded pebbles represented 22.4% of the total core weight. After contacting crushed core and aqueous solutions for various time periods, the samples were centrifuged, and the aqueous portion was filtered through 0.45 μm filters. The sample solids (crushed core) after centrifugation were composed of approximately 40.3% coarse sand, 4.2% fines, and 55.5% hydrated amorphous clay-like material.

Crushed core samples were also contacted with solutions of NaCl containing no alkaline additives to determine the amount of HCl required to titrate a blank sample. It was found that the pH of the blank increased with time, indicating that components of the sample slowly dissolved, increasing the sample alkalinity. The consumption measurements were corrected for the amount of alkalinity added by the rock.

The results of alkaline consumption measurements are given in Table 9. Without taking into account the increase in alkalinity of the blank, carbonate solution alkalinity has charged only 4% after 18 days. If corrections are made for the alkalinity of the blank, alkali consumption increased from 0.64 to 1.50 mg/g during the aging period of 31 days. The buffering effect of the reservoir rock and formation brine may alleviate alkalinity consumption in the reservoir caused by interaction with the rock. Other mechanisms of carbonate consumption, such as reaction with the oil and precipitation with divalent ions in the reservoir or makeup water, would still occur.

Alkali measurements were also made for effluent samples from several coreflood studies. Alkalinity consumption in Berea sandstone during a CJ2 surfactant ASP test (KMAY-2) was significantly less than the values reported for the static tests. With a contact time of approximately 1.5 days, alkalinity loss was 0.034 mg/g rock. This compares with uncorrected changes in alkalinity than with the corrected values. For an ASP coreflood in Karamay reservoir rock, Y1-1, the alkalinity loss was higher by a factor of 10, 0.3 mg/g, but still less than the lowest static value of 0.64 mg/g.

Table 9 Alkali Consumption with Karamay Reservoir Core

Sample description	Liquid/ solid ratio	Time, days	pH	Alkalinity, meq/mL (mg/mL)	Alkali consumed, mg/g	Alkali consumed, (total sample with pebbles) mg/g	Alkali consumed, % of total
Na_2CO_3 solution	-----	—	10.28	0.00931 (0.493)	—	—	—
Na_2CO_3 solution + crushed core	10:1	4	10.12	0.00916 (0.485)	0.83	0.64	16.9
Na_2CO_3 solution + crushed core	10:1	18	10.11	0.00894 (0.474)	1.49	1.15	30.3
Na_2CO_3 solution + crushed core	10:1	31	9.98	0.00914 (0.484)	1.93	1.50	39.3
NaCl solution + crushed core	10:1	3	7.31	0.00145 (0.077)	—	—	—
NaCl solution + crushed core	10:1	27	7.38	0.00351 (0.186)	-----	—	-----

Polymer Adsorption

Polymer adsorption was determined for samples prepared by contacting Karamay reservoir crushed core with polymer solutions prepared by dissolution of 0.5% NaCl in synthetic river water. The polymer was the polyacrylamide MO3000H-SF used in the coreflood experiments. The crushed rock was a mixture from core samples labeled Y43-3 (well # 2007), Y1-5 (well # 2007), and Y16-5 (well # 2004). Large pebbles of core were excluded from the sample of crushed material. The large pebbles that were excluded represented 50.4% of the total core weight.

Figure 20 shows the polymer adsorption isotherm. As equilibrium polymer concentration increased, adsorption increased to a plateau value of about 0.25 mg/g of crushed core. This value would be reduced by about 50% if corrected for the 50.4% of excluded pebbles, which should contribute very little to polymer adsorption. The value of 0.12 mg/g is relatively high, however.

Surfactant Adsorption

Surfactant adsorption experiments were conducted with alkali concentrations from 0.5% to 1.6% Na_2CO_3 concentration. The crushed rock, a mixture from core samples Y16-3, Y20-2, and Y20-1, was also used for the alkali consumption measurements and was described previously.

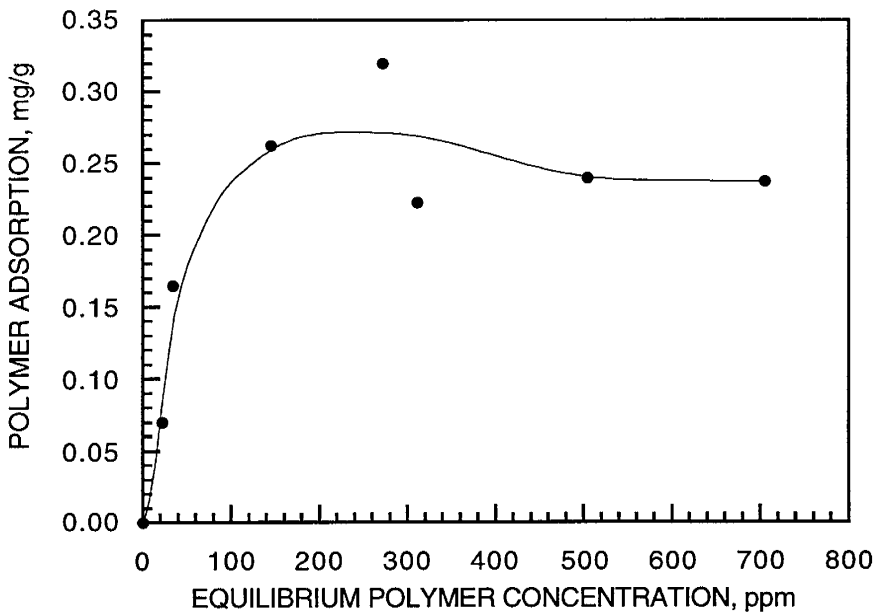

Figure 20 Polymer Adsorption as a Function of Polymer Concentration in Solution.

Adsorption isotherms for CJ-2 surfactant are shown in Figure 21. At 0.50, 1.00, and 1.60% Na_2CO_3 concentrations, surfactant adsorption exhibited maximums near 0.35% equilibrium CJ-2 concentration. Surfactant adsorption increased with increasing carbonate concentration. Increased surfactant adsorption at high Na_2CO_3 concentration is presumably due to increasing ionic strength. Similar results were previously reported for B-100 surfactant in the phase 1 report. Figure 22 shows a comparison of surfactant adsorption for B-100 and CJ-2 in 1% Na_2CO_3. Overall, adsorption is similar for the two surfactants. Peak adsorption for B-100 appears to be lower; however, this may be due to the different range of surfactant concentrations used for the B-100 experiments or to differences in the clay content of the rock samples used in the tests.

Figure 23 shows surfactant adsorption in the presence of polymer. The presence of 300 ppm of MO3000H-SF polymer did not change surfactant adsorption. Competitive adsorption between surfactant and polymer could reduce surfactant adsorption. However, surfactant-polymer interactions, including precipitation of material high in surfactant concentration, has been observed for these chemicals. For 0.2% surfactant concentration, two phases formed, and a polymer-surfactant complex appeared to be less dense than the aqueous fraction. At higher surfactant concentrations, the polymer-surfactant precipitated. For this study, the presence of some polymer in the surfactant solution showed no effect on surfactant loss.

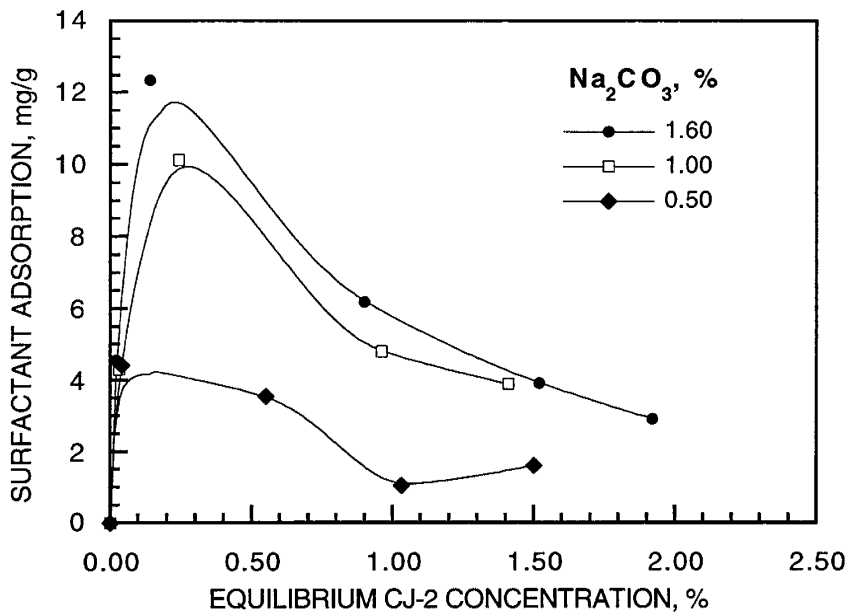

Figure 21 Static CJ2 Surfactant Loss Values on Crushed Karamay Reservoir Core.

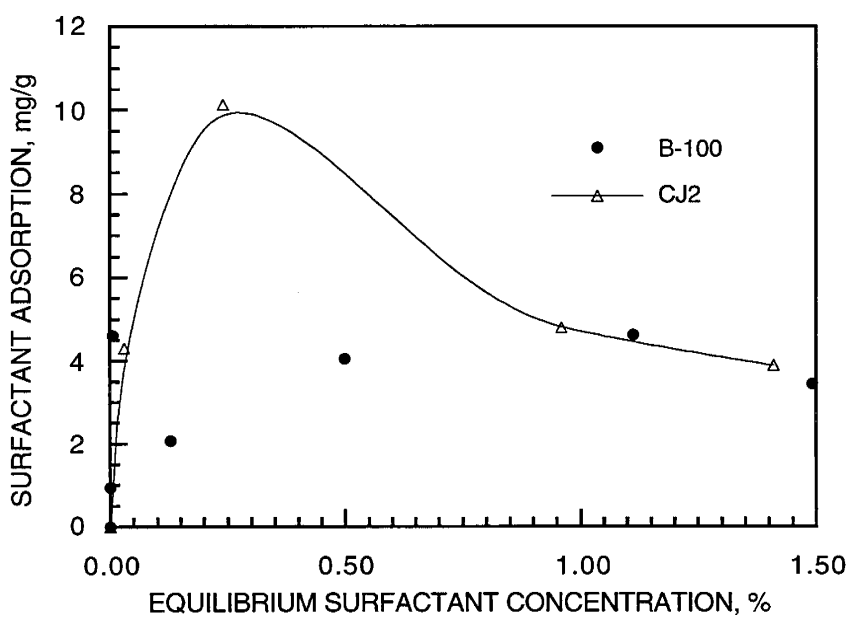

Figure 22 Static CJ2 and B-100 Surfactant Loss Values on Crushed Karamay Reservoir Core for Solutions Containing 1.0% Na_2CO_3.

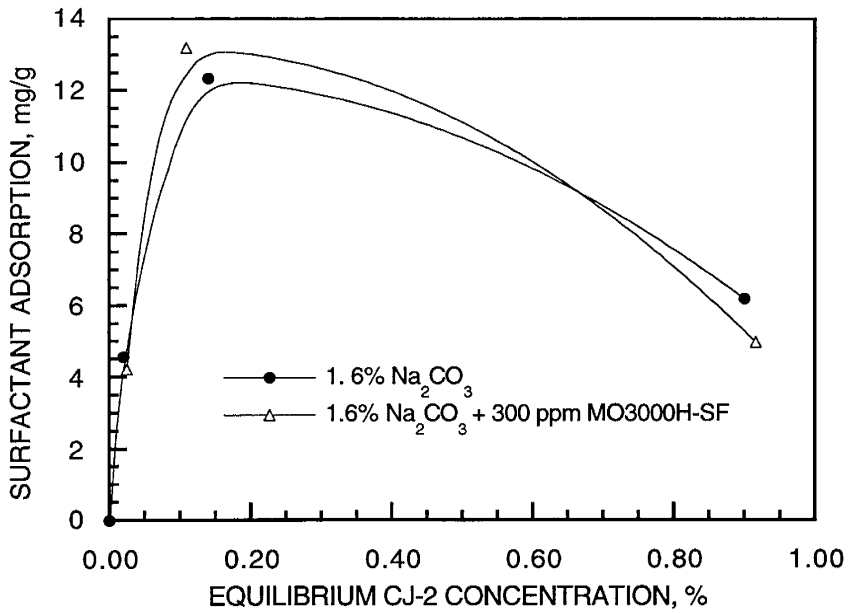

Figure 23 The Effect of the Presence of Polymer on CJ2 Surfactant Adsorption in 1.6% Na_2CO_3.

Surfactant concentrations in the coreflood effluents was used to calculate dynamic surfactant losses. Dynamic surfactant loss calculations were conducted for the CJ2 surfactant for both corefloods in Berea sandstone, KMAY-2 and KMAY-3. In addition, a chemical coreflood was conducted with the reservoir rock with the same formulation used in KMAY-3. This flood, KCF-5, used the reservoir core plug S42-2. The surfactant loss from this test was not significantly higher than that determined in the Berea corefloods tests and were much lower than the values obtained in the static tests. Results are summarized in Table 10.

Table 10 Surfactant Loss Under Dynamic Conditions for Coreflood Conducted in Berea Sandstone and Karamay Reservoir Core, KCF-5.

Sandstone Rock Type	Coreflood	Initial Anionic [Surfactant] wt %	Prepared [Na2CO3] in River Water wt %	Surfactant Loss mg/g rock
Berea	KMAY-2	5.0	1.6	0.17
Berea	KMAY-3	0.45	2.5	0.11
Reservoir	KCF-5	0.45	2.5	0.20

Additional results from dynamic tests, however, showed higher surfactant losses. Surfactant analysis of the core effluents was conducted to determine dynamic CJ2 surfactant loss values using

crushed Karamay reservoir rock. Figures 24 and 25 show the surfactant concentration curves for sandpack #1 and #2, respectively. Production of surfactant was delayed by approximately 0.25 PV in test #1 by the presence of polymer in the surfactant slug. Surfactant loss, however, was the same for both tests, 2.45 mg/g rock for test #1 and 2.50 mg/g rock for test #2. (These values have not been adjusted for the weight of the pebbles or fines excluded from the sample.) This is much higher than previously reported dynamic adsorption values for KCF-5 but are the same order of magnitude as the static adsorption values measured on crushed core samples.

The surfactant was exposed to a greater rock surface area in the sandpack tests than in the consolidated core experiments. The surface area in the sandpack tests would also more closely resemble that of the crushed rock used for the static adsorption experiments. Higher chemical adsorption than the consolidated core experiment can therefore be expected.

The effluent from Karamay reservoir core Y1-1 coreflood tests was also examined for presence of surfactant. In this case, no surfactant was detected in the effluent samples. Only the aqueous samples were tested for surfactant. No attempt was made to extract surfactant from the oil. If surfactant and oil are produced together, however, emulsions are usually observed. No emulsions were produced during this experiment. Based on the amount of surfactant injected, adsorption calculation would indicate a loss of 1.1 mg/g rock. This value is five times greater than the value measured for S42-2 and approaches the dynamic values obtained in sandpacks and the static adsorption values.

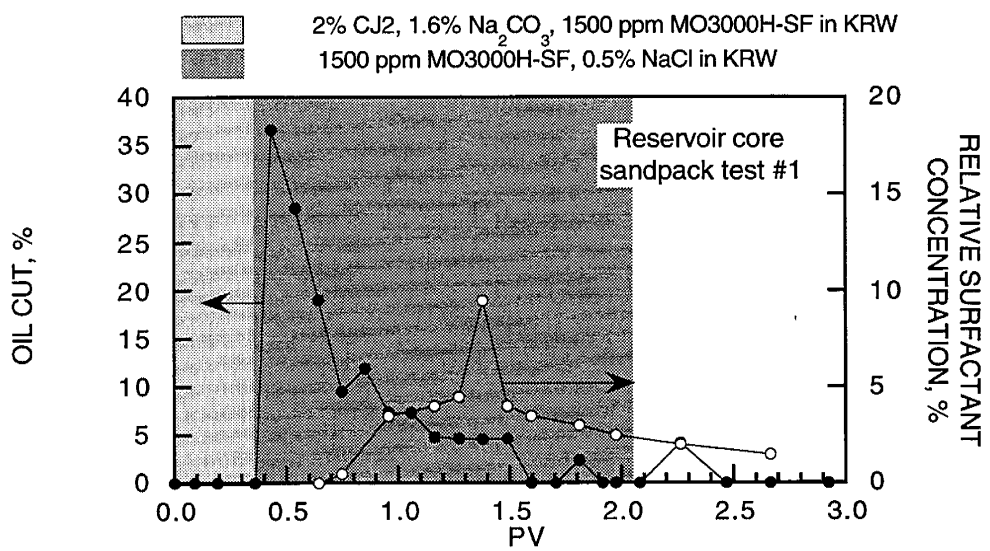

Figure 24 Oil Cut and Surfactant Concentration Relative to the Injected Surfactant Concentration for Sandpack Test #1.

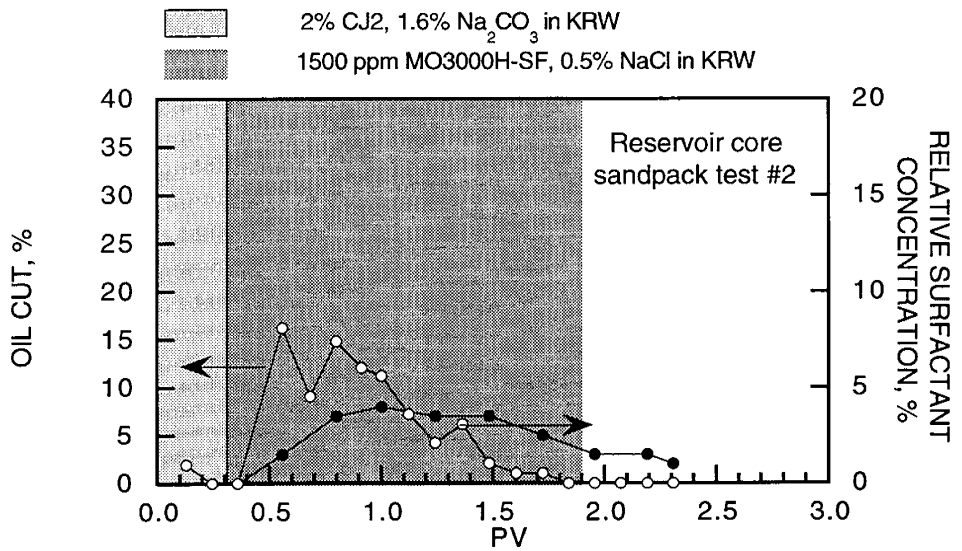

Figure 25 Oil Cut and Surfactant Concentration Relative to the Injected Surfactant Concentration for Sandpack Test #2.

These values are in the range but somewhat higher than values reported in the literature for static and dynamic surfactant losses.[1] [2] Static adsorption results often measure in the 1 to 5 mg/g range, while dynamic test values may be lower by a factor of ten. Comparison of results is most effective when comparative rock surface area, cation exchange capacity (CEC), or clay content is known.

In summary, adsorption losses in most cases were high. Chemical losses could be significant, and to maximize oil production, chemical design should not minimize chemical slug components. Better characterization of the producing reservoir rock to determine surface area and clay content would improve prediction of chemical losses in the reservoir.

Steady-State Relative Permeability

An imbibition steady-state relative permeability test was conducted using Karamay core plug Y2-1 (well # ES2004, depth 651.79 m). Average fluid saturations during the experiment were measured using X-ray monitoring techniques. Pressure measurements were made over a 2.5 cm distance in the middle of the core to avoid measuring end effects. Initially, the core was saturated with oil and permeability to oil was measured as 26.5 mD. The core was then cleaned before use in

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[1],Hawkins, B., K. Taylor, and H. Nasr-El-Din, Mechanisms of Surfactant and Polymer Flooding: Application to David Lloydminster and Wainwright Sparky Fields. Peteroleum Society of CIM and AOSTRa, Paper CIM/AOSTRA 91-28. pp28-1 to 28-16.

[2]Baviere, M., P. Glenat, V. Plazanet and J. Labrid. "Improvement of the Efficiency/Cost ratio of Chemical EOR Processes by Using Surfactant, Polymers, and Alkalis in Combination." SPE/DOE 27821, presented at the SPE/DOE Ninth IOR Symposion, Tulsa, Oklahoma, April 17-20, 1994.

distance in the middle of the core to avoid measuring end effects. Initially, the core was saturated with oil and permeability to oil was measured as 26.5 mD. The core was then cleaned before use in the steady-state experiment. After saturation with brine, the absolute permeability of the plug to brine was measured as 24.2 mD. The sample was flooded with more than 30 pore volumes of oil. Oil injection rates as high as 60 mL/hr were used to reach a residual brine saturation. X-ray measurements indicated a residual brine saturation of 34.5%. Volumetric measurements (with pore volume corrected for squeeze-out) indicated a brine saturation of about 33%. The permeability to oil at residual brine was measured as 19.8 mD.

Steady-state relative permeability results for the imbibition cycle are shown in Figure 26. The oil relative permeability for sample Y2-1 compares with the oil relative permeability for Berea sandstone used for most of the oil recovery experiments in this study. The water relative permeability for Y2-1 was higher than that measured for the Berea sandstone.

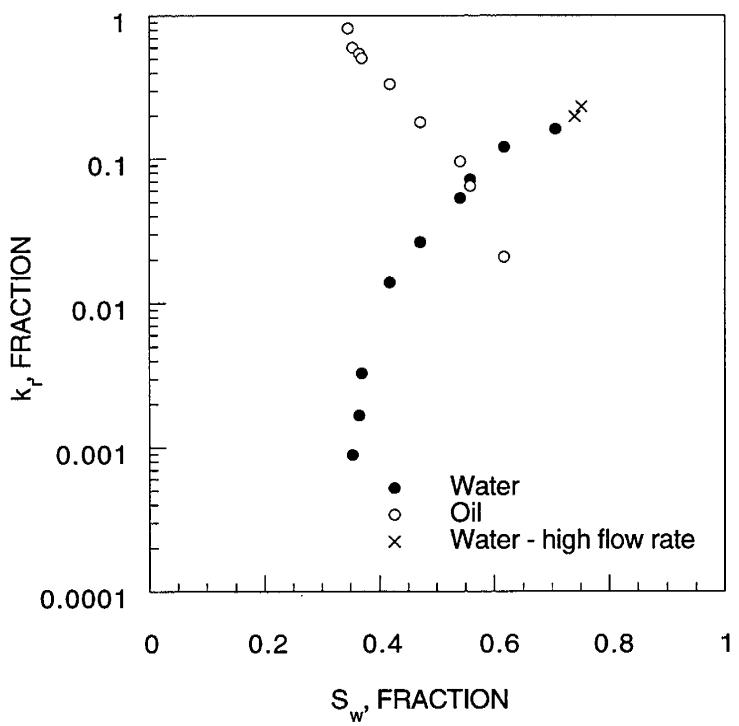


Figure 26 Sample Y2-1 Steady-State Imbibition Relative Permeability Results.

SUMMARY AND CONCLUSIONS

A laboratory research program for the Xinjiang Petroleum Administration (XPA) was conducted to evaluate alkaline-surfactant-polymer chemical systems for the Karamay reservoir. Results and conclusions includes the following:

- Steady-state relative permeability curves were measured for one Karamay reservoir core plug.

- Karamay reservoir rock was sensitive to low-salinity water. Exposure to Karamay river water could result in a significant reduction (90% reduction or more) in liquid permeability. In addition, core that was initially saturated with a nondamaging brine (3% KCl) was reduced in permeability after exposure to Karamay formation water (50%–80% reduction).

- Brine sensitivity tests with Karamay reservoir core indicated that no significant permeability reduction was observed when a solution of 3% NaCl in Karamay river water was injected. Reduction in brine salinity to 2% NaCl also caused little change in permeability. Reduction in brine salinity to 1.5% NaCl, however, resulted in a slow decline in liquid permeability. Injecting lower salinity water is not recommended for Karamay formation rock.

- Alkaline-surfactant-polymer chemical systems could be designed that generate very low interfacial tensions with Karamay crude oil. Sodium carbonate appeared to be the most effective alkaline agent. Low IFT systems included alkyl aryl sulfonates such as Petrostep B-100 and petroleum sulfonates such as CJ2 supplied by the XPA. The systems generated low IFTs over a fairly broad range of alkaline (Na_2CO_3) concentrations.

- Because of the water sensitivity of the core, some effort was undertaken to design a chemical system for higher salinity and alkalinity ranges. The low IFT region of either B-100 or CJ2 can be adjusted for different salinity ranges by addition of small amounts (0.08% or less) of nonionic or anionic ethoxylated surfactants. If the salinity of a preflush or chemical slug is increased to prevent formation damage caused by water sensitivity, the chemical slug composition can be adjusted to maintain good IFT activity.

- Low IFTs are necessary but not sufficient to produce the maximum amount of oil during coreflood recovery tests using both Berea sandstone and reservoir core. For corefloods conducted in Berea sandstone core, an alkaline-polymer slug produced as much or more oil than ASP formulations where the surfactant concentration was 0.5%. In reservoir core, however, the AP was not as successful as tests conducted with ASP formulations.

- In Berea sandstone, only formulations with higher surfactant concentration (1% or more) produced more oil than the AP test. Injecting excessive amounts of surfactant, however, caused production of oil and water emulsions. The emulsions produced during coreflood experiments were stable and required special treatment to break.

- For tests conducted in both Berea sandstone and Karamay reservoir core (plugs and sandpacks), oil saturations were reduced to similar levels (< 10% S_{orc}) using the 2% CJ2-1.6% Na_2CO_3 chemical system. Chemical systems designed for higher salinity (2.5% Na_2CO_3) were slightly more successful in reducing oil saturation in reservoir core than in Berea sandstone core (14%–16% S_{orc} versus 23%–25%). Recovery efficiencies for the CJ2-2.5% Na_2CO_3 system were very similar for reservoir core and Berea sandstone (42% and 39% of S_{row}), however.

- In general, the CJ2 surfactant is as effective as B-100 in recovering oil. CJ2 is available locally (in China).

- An oil recovery experiment using stacked reservoir was not successful. Because of the difficulty in obtaining squared ends on these cores, poor capillary contact existed between cores. Packing the space between cores with crushed reservoir rock did not improve fluid continuity. Oil saturations remained high between the reservoir rocks after both waterflood and chemical flood. CT-imaging was helpful in illustrating the problems associated with this test. Because of these difficulties, stacked core experiments should be avoided in the laboratory.

- Poor quality cores (low permeability and poor consolidation in higher permeability cores) received at NIPER necessitated extra effort and/or repetition of tests, resulting in a delay in completing the project.

- Cation exchange capacity (CEC) values were high for the reservoir cores (0.04 to 0.11 meq/g as reported in the Phase 1 Report). The core contained significant amounts of clay or other small particle size (high surface area) material. Chemical adsorption would be a problem in areas of the reservoir represented by these core samples. This may partially explain the requirement to increase surfactant concentration to make an effective chemical system for the Karamay reservoir.

- Static adsorption of surfactant and polymer on Karamay reservoir rock was also high. Maximum surfactant adsorption occurred at 0.5 wt% and declined as surfactant concentration increased. The presence of polymer did not affect the adsorption characteristics of the surfactant. Dynamic adsorption measurements using crushed reservoir rock compared with the

static adsorption results. Dynamic adsorption measurements using consolidated reservoir rock, in most cases, were less than the static adsorption values. Results varied, however, so that chemical losses should not be underestimated when designing the chemical system for field use.

- Adverse surfactant-polymer interactions were observed in the laboratory. In the field, chemical slugs should not be prepared and allowed to remain static to prevent CJ2 or CJ2-polyacrylamide material precipitation. Injectivity around the wellbore may be caused by chemical interaction products.

RECOMMENDATIONS

- An ASP process can be applied in the Karamay reservoir provided the operators are cognizant of and design for the problems associated with water sensitivity and adsorption problems of the reservoir rock. Every attempt should be made to characterize the actual reservoir rock.

- The AP system should contain at least 1% and preferable 2% CJ2 with 1.6% Na_2CO_3. At least a 1500 ppm polymer containing 0.5% NaCl should be used in the mobility control buffer.

- The slug design should not minimize slug size and chemical concentrations because of the problems observed with the reservoir rock. This especially true for the pilot test.

- Use a preflush or add salt to chemical slugs that are formulated with Karamay river water to minimize formation damage from injection of fresh water. Preflush salinity should be the equivalent of 1.5% NaCl or greater with a preferred preflush of 2% or greater.

- Run confirmation tests and maintain a quality control program to check the effectiveness of the final chemical products that are used in the field test. This includes IFT, viscosity, and surfactant activity measurements.

- In the field, inject at least some surfactant slug (i.e. for at least a week) without polymer to monitor injectivity of AS slug. If injectivity problems occur either before or after mixing polymer into the chemical slug, it would be easier to diagnose and correct for the problem.

- Prior experience at NIPER indicated that addition of a very small quantity ($\approx$0.1%) of crude oil or refined oil alleviated polymer-surfactant interaction problems. This should be tested with the CJ2 surfactant system.

APPENDIX 1

KARAMAY CORE DESCRIPTION

Core plugs 3.8 cm. in diameter and approximately 5 to 8 cm long were received from Xinjiang Petroleum Administration (XPA) for use in the Karamay ASP evaluation project. A core identification list is attached. Core quality was relatively poor. A number of pieces contained large pebbles. Many core plugs were of low permeability. A number of other cores were friable and disintegrated on handling. Poor quality core increased the time required to obtain oil recovery results using reservoir core and affected interpretation of oil recovery results. Preparing these cores to have square ends for use in coreholders was often very difficult. Cleaning core often made them more difficult to handle. For friable cores, the best procedure was to freeze the core using liquid nitrogen and wrap with Teflon tape and shrink tubing to protect the edges. Shrink tubing, however, made the cores too large for some of our coreholders and could not be used in all cases. Figure A1-1 shows pictures of some of the cores including those with large pebbles (S57-3, Y27-2, S16-3), low permeability (Y20-1), uneven ends (Y27-2), and relatively uniform, high permeability but friable core (Y2-2). Figure A1-2 shows size distribution of crushed core material that was used for sandpack oil recovery tests.

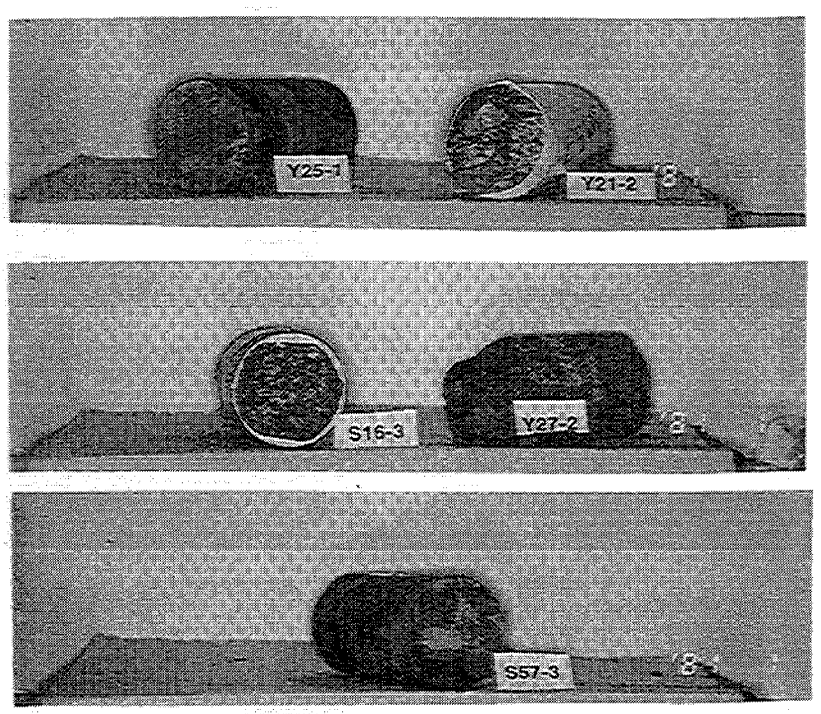


Figure A1-1 Pictures of Karamay Core Plugs Showing Some of the Problems Associated with the Core.

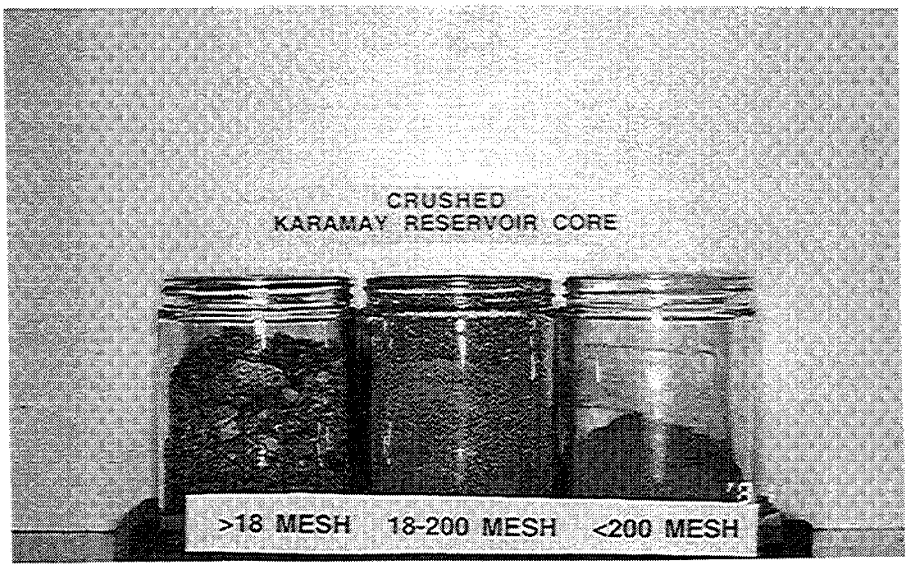


Figure A1-2 Size Variation in Crushed Karamay Core that was Used for Sandpack Oil Recovery Tests.

CORE SAMPLE LIST

Middle District No. 2 Well No. ES2004

Number	Field Sample Number	Sample Length (cm)	Formation	Interval (m)	Length / Footage	Lab. Sample Number	Depth (m)	Lithological and Oil-bearing Character		
								Lithological	Cement	Oil-bearing
1	Y1	10	T_2^1	649.79-652.33	2.54/2.54	Y1-1	650.94	Coarse sand	Medium	Stained
			T_2^1		2.54/2.54	Y1-2	650.99	Coarse sand	Medium	Stained
2	Y2	12	T_2^1	649.79-652.33	2.54/2.54	Y2-1	651.79	Coarse sand	Medium	Spotted
			T_2^1		2.54/2.54	Y2-2	651.83	Coarse sand	Loose	Spotted
3	Y8	13	T_2^1	658.76-662.76	2.53/4.0	Y8-1	659.53	Conglomerate	Loose	Stained
			T_2^1		2.53/4.0	Y8-2	659.58	Conglomerate	Loose	Stained
4	Y13	7	T_2^1	665.77-666.65	0.88/0.88	Y13-1	666.19	Conglomerate	Loose	Spotted
5	Y17	11	T_2^1	669.73-671.73	2.2/2.2	Y17-1	670.72	Conglomerate	Loose	Spotted
6	Y18	14	T_2^1	669.53-671.73	2.2/2.2	Y18-1	671.14	Conglomerate	Loose	Spotted
			T_2^1		2.2/2.2	Y18-2	671.19	Conglomerate	Loose	Spotted
7	Y20	9	T_2^1	671.73-673.60	1.87/1.87	Y20-1	673.20	Conglomerate	Medium	Spotted
8	Y23	11	T_2^1	673.60-674.85	1.25/1.25	Y23-1	674.00	Conglomerate	Medium	Spotted
			T_2^1		1.25/1.25	Y23-2	674.05	Conglomerate	Medium	Spotted
9	Y24	13	T_2^1	673.60-674.85	1.25/1.25	Y24-1	674.67	Conglomerate	Medium	Spotted
			T_2^1		1.25/1.25	Y24-2	674.72	Conglomerate	Medium	Spotted
			T_2^1		1.25/1.25	Y24-3	674.76	Conglomerate	Medium	Spotted
			T_2^1		1.25/1.25	Y24-4	674.81	Conglomerate	Medium	Spotted
10	Y25	12	T_2^1	674.85-678.15	3.3/3.3	Y25-1	675.14	Conglomerate	Medium	Spotted
			T_2^1		3.3/3.3	Y25-2	675.19	Conglomerate	Medium	Spotted

CORE SAMPLE LISTS

Middle District No. 2 Well No. ES2007

Number	Field Sample Number	Sample Length (cm)	Formation	Interval (m)	Length / Footage	Lab. Sample Number	Depth (m)	Lithological and Oil-bearing Character		
								Lithological	Cement	Oil-bearing
1	Y1	24	T_2^1	643.00-647.05	4.05/4.05	Y1-2	646.05	Pebble sand stone	Medium	Stained
			T_2^1			Y1-3	646.09	Pebble sand stone	Medium	Stained
			T_2^1			Y1-4	646.13	Pebble sand stone	Medium	Stained
			T_2^1			Y1-5	646.18	Pebble sand stone	Medium	Stained
2	Y2	21	T_2^1	643.00-647.05	4.05/4.05	Y2-1	646.33	Conglomerate	Medium	Stained
3	Y4	14	T_2^1	647.05-652.00	4.95/4.95	Y4-1	649.55	Fine sand stone	Loose	Spotted
4	Y13	15	T_2^1	647.05-652.00	4.95/4.95	Y13-3	650.89	Fine sand stone	Loose	Spotted
5	Y16	25	T_2^1	647.05-652.00	4.95/4.95	Y16-2	651.64	Medium fine sand stone	Loose	Spotted
			T_2^1			Y16-3	651.70	Medium fine sand stone	Loose	Spotted
			T_2^1			Y16-4	651.74	Medium fine sand stone	Loose	Spotted
			T_2^1			Y16-5	651.79	Medium fine sand stone	Loose	Spotted
6	Y18	19	T_2^1	655.92-657.86	1.9/1.94	Y18-2	656.73	Conglomerate	Medium	Spotted
7	Y19	14	T_2^1	655.92-657.86	1.9/1.94	Y19-1	656.84	Pebble sand stone	Medium	Stained
			T_2^1			Y19-2	656.88	Pebble sand stone	Medium	Stained
			T_2^1			Y19-3	656.93	Pebble sand stone	Medium	Stained
8	Y20	33	T_2^1	655.92-657.86	1.9/1.94	Y20-1	657.37	Pebble sand stone	Medium	Stained
			T_2^1			Y20-2	657.32	Pebble sand stone	Medium	Stained
			T_2^1			Y20-3	657.25	Pebble sand stone	Medium	Stained
9	Y21	14	T_2^1	[illegible]	[illegible]	Y21-2	[illegible]	Conglomerate	Medium loose	Spotted
10	Y23	11	T_2^1	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
11	Y24	12	T_2^1	[illegible]	[illegible]	Y24-2	661.0	Conglomerate	Loose	Spotted

CORE SAMPLE LISTS

Middle District No. 2 Well No. ES2007

Number	Field Sample Number	Sample Length (cm)	Formation	Interval (m)	Length / Footage	Lab. Smaple Number	Depth (m)	Lithological and Oil-bearing Character		
								Lithological	Cement	Oil-bearing
12	Y26	20	T_2^1	666.2-667.83	1.61/1.63	Y26-1	666.32	Conglomerate	Loose	Stained
			T_2^1			Y26-2	666.37	Cnglomerate	Loose	Stained
			T_2^1			Y26-3	666.28	Conglomerate	Loose	Stained
			T_2^1			Y26-4	666.23	Conglomerate	Loose	Stained
13	Y27	14	T_2^1	666.2-667.83	1.61/1.63	Y27-1	667.13	Conglomerate	Loose	Stained
			T_2^1			Y27-2	667.18	Conglomerate	Loose	Stained
14	Y30	9	T_2^1	667.33-670.48	2.60/2.65	Y30-1	668.12	Conglomerate	Loose	Stained
15	Y31	14	T_2^1	667.33-670.48	2.60/2.65	Y31-1	669.23	Conglomerate	Loose	Stained
16	Y37	14	T_2^1	670.48-672.85	2.37/2.37	Y37-1	671.53	Conglomerate	Medium	Stained
17	Y43	17	T_2^1	672.85-675.4	2.55/2.55	Y43-2	673.86	Conglomerate	Medium loose	Stained
			T_2^1			Y43-3	673.91	Conglomerate	Medium loose	Stained
18	S5	12	T_2^1	643.0-647.05	4.05/4.05	S5-2	646.53	Fine conglomerate	Medium	Stained
19	S16	25	T_2^1	647.05-652.00	4.95/4.95	S16-3	649.22	Fine sand stone	Loose	Oil sand
			T_2^1			S16-4	649.28	Fine sand stone	Loose	Oil sand
20	S42	21	T_2^1	663.91-665.35	1.33/1.44	S42-2	664.36	Pebble sand stone	Loose	Spotter
			T_2^1			S42-3	664.40	Pebble sand stone	Loose	Spotter
21	S48	14	T_2^1	665.35-666.2	0.85/0.85	S48-1	665.81	Conglomerate	Medium loose	Stained
			T_2^1			S48-2	665.0	Conglomerate	Medium loose	Stained
22	S57	21	T_2^1	667.22-670.48	2.31/2.65	S57-3	668.92	Conglomerate	Loose	Stained
23	S64	15	T_2^1	672.05-675.4	2.31/2.55	S64-4	673.11	Conglomerate	Medium loose	Stained

APPENDIX 2

EXPERIMENTAL PROCEDURES

Coreflooding procedures

Oil recovery experiments were conducted in Hassler-sleeve core holders. Core effluents were collected using a fraction collector. Pressure differentials across the core and water and oil production was measured for each test. For selected tests, effluent samples were analyzed for production of surfactant, polymer, and selected ions. Dynamic chemical loss values were calculated based on measurements of produced fluid components. Figure A2-1 shows a representation of the coreflooding equipment, and Table A2-1 summarizes the sequence of coreflooding procedures used in this study.

CT-imaging

A Siemens Somatron II CT scanner was used to image the dry and fluid-saturated cores in the high resolution mode of 125 kV and 460 mAs. Each scan produced an image 256 x 256 volume elements (voxels) that represented a core volume of 0.5 x 0.5 x 8 mm. The images were stored temporarily on the CT scanner hard disk and then transferred to other computer storage for further examination or data manipulation.

Figure A2-1 Schematic Representation of Coreflooding Equipment.

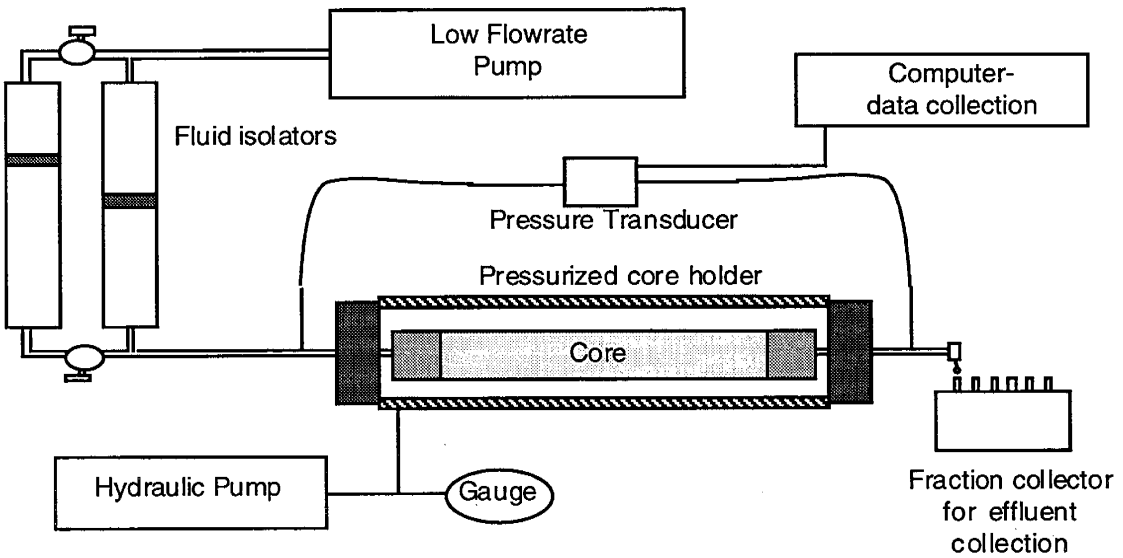


Table A2-1 Coreflooding Procedures Including CT-Imaging Operations

Experimental operation	Measurement	Calculation	CT Scan
Select, trim, clean (as required) and dry core	Measure core dimensions		
Place core in coreholder (aluminum for CT test), check for leaks, and apply overburden pressure	Measure dry weight		Scan dry core
Evacuate, flush with CO_2, and saturate with degassed 3% KCl followed by formation brine	Measure saturated weight	Calculate pore volume	Scan saturated core
Flow formation oil to residual brine saturation (tagged oil for CT test)	Measure brine out	Calculated oil saturation at residual water saturation	Scan oil-saturated core
Waterflood to residual oil saturation	Measure oil out	Calculate residual oil saturation	Scan waterflooded core
Inject chemical slugs: Preflush ASP Polymer postflush brine	Measure oil and brine production and pressure	Calculate pore volume, oil cut, changes in residual saturation, accumulated oil production, etc.	Scan several times as needed to monitor oil advance
Collect effluent samples	Analytical measurements on effluent samples as needed: Viscosity pH and alkalinity Surfactant and polymer concentrations Ion concentrations, etc.	Determine effluent profiles. Calculate surfactant and polymer losses, etc.	

Calculations and Image Processing

To determine porosity and fluid saturations, each scan must be conducted on the same area of the core under the same scanning conditions. A computer-controlled positioning table allows the core to be repositioned in the beam within 0.012 mm. In addition, scans were made during different stages of the coreflood. After X-ray scanning of a core, the produced images are stored for further viewing and analysis to determine porosity and two-phase fluid saturation distributions. The individual image files can then be merged to produce one file for 3-D image viewing. The 3-D reconstruction of the core can then be rotated and sliced for viewing along any of the *XY*, *XZ*, and *YZ* planes through the core.

Static Adsorption Measurements

Static tests were conducted using crushed Karamay reservoir rock to determine chemical consumption values for alkali, surfactant, and polymer, as well as surfactant in the presence of polymer.

Alkali consumption measurements were determined for samples prepared by contacting crushed Karamay reservoir rock with 0.01N Na_2CO_3 (approx. 0.053% Na_2CO_3). The crushed rock was a mixture from core samples labeled Y16-3, Y20-2, and Y20-1. Large pebbles within the core were excluded from the sample of crushed material. The excluded pebbles represented 22.4% of the total core weight. The crushed rock used in the adsorption test were composed of approximately 40.3% coarse sand, 4.2% fines, and 55.5% hydrated amorphous clay-like material. After contacting crushed rock and aqueous solutions for various time periods, the samples were centrifuged, and the aqueous portion was filtered through 0.45 μm filters. Total alkalinity was determined by titrating the aqueous filtrates to pH 4.0 with 0.01N HCl. Carbon dioxide was excluded from the samples as much as possible, and samples were gently agitated while aging.

Crushed core samples were also contacted with solutions of NaCl containing no alkaline additives to determine the amount of HCl required to titrate a blank sample. It was found that the pH of the blank sample increased with time, indicating that components of the sample slowly dissolved, increasing the sample alkalinity. The consumption measurements were corrected for the amount of alkalinity added by the rock. This added alkalinity, however, may help buffer the chemical slug. Alkaline consumption may be minimized by this reservoir characteristic.

Surfactant adsorption experiments were conducted with alkali concentrations from 0.5% to 1.6% Na_2CO_3 concentration. The crushed core was a mixture from core samples Y16-3, Y20-2, and Y20-1, which was also used for the alkali consumption measurements and was described previously. The experiments were conducted at a liquid/solid ratio of 2.5, and surfactant analyses were performed after 24 hours of contact time. Simulated Karamay river water was used for solution preparation. Surfactant adsorption in the presence of polymer was also evaluated for one alkali concentration. Epton two-phase methylene blue method was used for surfactant analyses.

Polymer adsorption was determined for samples prepared by contacting Karamay reservoir crushed core with polymer solutions prepared by dissolution of 0.5% NaCl in synthetic river water. The polymer selected for the experiments was MO3000H-SF, a polyacrylamide polymer manufactured in Japan. The crushed core was a mixture from core samples labeled as Y43-3 (well 2007), Y1-5 (well 2007), and Y16-5 (well 2004). Large pebbles of core were excluded from the sample of crushed material. The excluded pebbles represented 50.4% of the total core weight.

Formation Damage in Karamay Reservoir Core

Change of permeability to different salt solutions was determined for a sample of Karamay reservoir core, S42-2. Table A2-2 summarizes core properties for the sample used in this test.

A tracer test using 10% 2-propanol (IPA) in 3% KCl was conducted to determine if the core contained significant heterogeneities that would affect fluid contact and flow characteristics. Formation brine was used to flush the tracer solution through the core. Alcohol was analyzed by gas chromatography. A small amount of tracer broke through at approximately 0.7 PV. However, the major breakthrough of tracer occurred at 1 PV, which indicated that the core was relatively uniform and contained no major high permeability streaks.

After injection of formation brine, a series of aqueous solutions were injected into the core. In order of injection, these solutions included Karamay river water, 3% KCl, 2% NaCl, 5% KCl, and 3% NaCl. Changes in fluid permeability were monitored by measuring the fluid pressure drop across the core.

Steady-State Relative Permeability

Steady-state relative permeability measurements were conducted on Karamay core plug Y2-1 (well # ES2004, depth 651.79 m) using X-ray measurement techniques to determine average saturations along the core during flow of various ratios of oil and brine.

Test fluids included a filtered 17.7 cP crude oil, brine, and tagged brine. The brine consisted of 3 wt% KCl in deionized water. The tagged brine was prepared with 3 wt% KCl and 7 wt% KI in deionized water. The tagging agent (KI) was added to the brine to enhance its X-ray absorption characteristics. The tagged brine viscosity was 1.03 cP. The X-ray absorption properties of the three fluids were measured with the X-ray generator set at 55 kV tube potential and 20 mA tube current.

Sample Y2-1 was trimmed to provide flat, parallel ends that are perpendicular to the long axis of the plug. The edges of the ends chipped during the trimming process. These chipped areas were

Table A2-2 Core properties for Permeability Damage Test with Karamay Reservoir Rock

Core I.D.	Depth, m	Core length and diameter, cm	Porosity, %	Permeability to 3% KCl brine, mD	Comments
S42-2	664.36	7.254 x 3.8	23.1	397	Loose, pebble sandstone

filled with a zirconia cement so that the ends would be in good contact with coreholder end-pieces. The resulting sample length was approximately 6.0 cm. The pore volume of the unstressed sample was approximately about 11 cm^3, yielding a porosity of approximately 16%. The sample was saturated within a pressure vessel in which the sample was unconfined. This was accomplished by first evacuating the pressure vessel to a low vacuum pressure. Next, the vessel was filled with brine and then pressurized to 500 psig

The sample was transferred from the saturation vessel to a coreholder. The coreholder featured side pressure taps to permit measurements of pressure drops along the length of the sample rather than from end to end. Displaced fluid was measured as the confining pressure was applied. Measurements were recorded at several confining pressures between 100 and 750 psig. The system was allowed to equilibrate approximately 10 minutes between measurements. The compressibility measurement shown in Figure A2-2 represents the change in pore volume for this core under laboratory confining pressure conditions and is not representative of compressibility under reservoir conditions. Change in pore volume for other Karamay cores would depend on mineral composition, presence of large pebbles, and cementation strength.

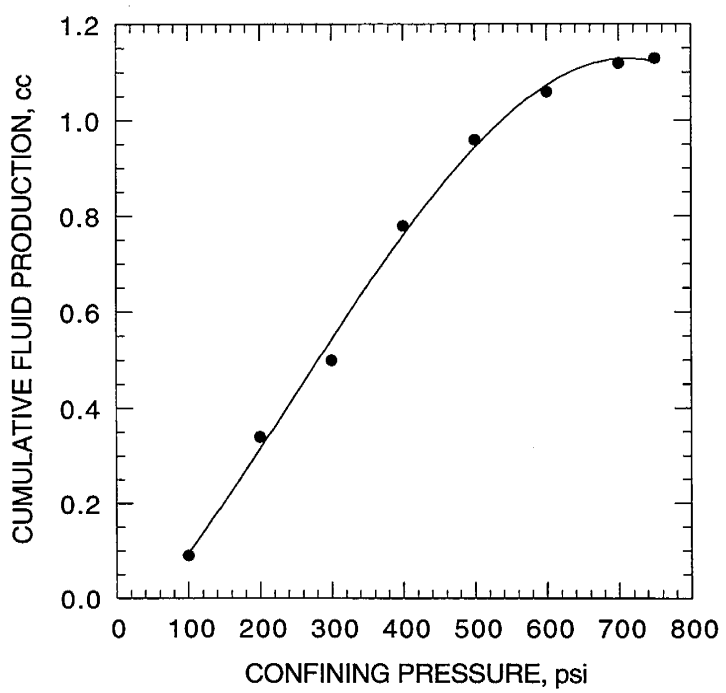


Figure A2-2 Sample Y2-1 Water Volume Produced by Increasing Confining Stress.

The coreholder with sample Y2-1 was placed on the X-ray table. The long axis of the plug was oriented horizontally. The sample was X-ray scanned at multiple positions along its length. The sample was then flooded with more than 10 pore volumes of tagged brine and scanned again. These scans along with the X-ray absorption properties already measured of the oil, brine, and tagged brine were used in the X-ray calibration of the sample. All subsequent flow tests were conducted using the oil and tagged brine.

Raw scan data of the rock saturated with brine and with tagged brine is shown in Figure A2-3. The relative values of the data are representative of sample density (the smaller the value, the larger the density).

The absolute permeability of the plug to brine was measured as 24.2 mD. The sample was flooded with more than 30 pore volumes of oil. Oil injection rates as high as 60 mL/hr were used to reach a residual brine saturation. X-ray measurements indicated a residual brine saturation of 34.5%. Volumetric measurements (with pore volume corrected for squeeze-out) indicated a brine saturation of about 33%. The permeability to oil at residual brine was measured as 19.8 mD. A steady-state imbibition relative permeability test was performed with brine fractional flows of 0.025, 0.05, 0.1, 0.4, 0.7, 0.9, 0.95, 0.99, and 1. More than nine total pore volumes of fluid were allowed to flow through the sample at 30 mL/hr at each fractional flow rate. End-point brine permeability and saturation measurements were recorded with brine flowrates of 30, 60, and 120 mL/hr to investigate the dependence of residual oil saturation on flow rate.

Throughout the test, pressure drops were measured across a 2.54 cm length near the middle of the sample. This was done in an attempt to prevent capillary end effects from influencing relative permeability results. The saturation data indicate that saturations varied across the measurement length even with steady-state flow conditions. Figure A2-4 illustrates this variance for three steady-state conditions. Steady-state relative permeability curves for an imbibition cycle is presented in the Results section.

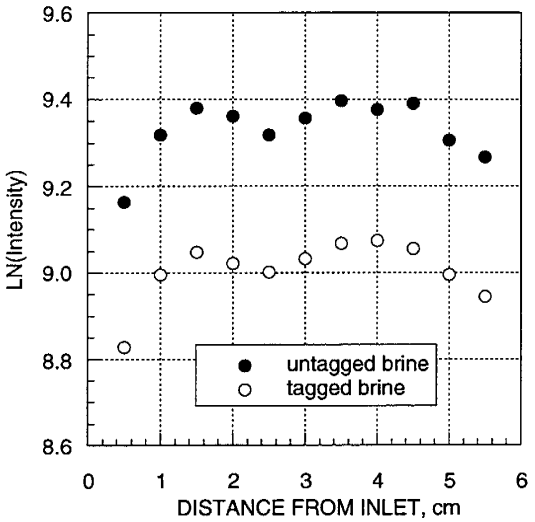


Figure A2-3 X-ray Data from Scans of Sample Y2-1 when the Plug was Saturated with Untagged and Tagged Brines.

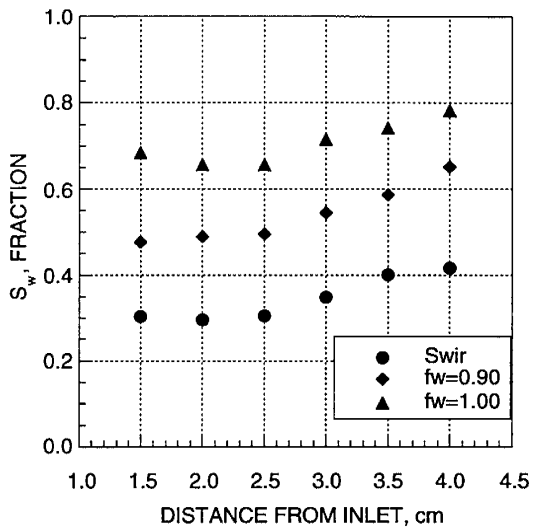


Figure A2-4 Sample Y2-1 Saturation Profiles, where S_{wir} = Irreducible Water Saturation and f_w = Water Fractional Flow

Analysis Procedures

The Phase 1 included procedures for a number of analytical operations used during this study. Two additional analytical procedures were utilized during Phase 2. The procedures to determine alkalinity and polymer analysis are attached.

SOP No.	: CHEMQA-XX
Revision No.	: 0
Date	: March 28, 1995
Page	: 1 of 2
Prepared by	: T. FRENCH
ES&H	:
Manager	:
Director	:

METHOD FOR DETERMINATION OF CARBONATE ALKALINITY

The following method is used specifically to determine the total alkalinity of aqueous solutions that contain carbonates.

Assumption: sample solution density is 1 and that weighing the sample is inherently more accurate than the sample volume measurement.

The endpoint for titration of carbonates with HCl is pH 4.0. Since it is difficult to end the titration at exactly pH 4.0, a correction is made for over-titration.

1. Calibrate pH meter according to manufacturers instructions.
2. Tare 5 mLplastic test tube.
3. Aliquot 1 mL of sample into test tube.
4. Record sample weight.
5. Rinse electrode with deionized water.
6. Blot excess water from electrode.
7. Measure pH of sample.
8. Remove electrode from sample, leaving as little sample liquid on electrode as possible.
9. Do not blot electrode, but place electrode in a position that will allow the sample liquid to remain on electrode.
10. Begin titration by adding an aliquot of standardized acid (usually 0.2N HCl) from a microburet.
11. Mix sample and acid for about 30 sec. with Vortex mixer.
12. Measure pH of sample.
13. Repeat acid additions (Steps 10 through 12) until pH is equal to or slightly below pH 4.
14. Record final pH and total volume of acid added.

Calculations:

Alkalinity, meq/mL = [(Acid Volume, mL) (Acid Normality, meq/mL) - (Sample Volume + Acid Volume, mL) ($10^{-\text{Final pH}}$ - 0.0001)] /(Sample Volume, mL)

The terms (Sample Volume + Acid Volume, mL) ($10^{-\text{Final pH}}$ - 0.0001) is the correction for over-titration past pH 4.0.

Sample calculation:

Sample weight = 1.002 gm
Acid Normality = 0.2 meq/mL
Initial pH = 9.34
Final pH = 3.42
Acid volume = 0.86 mL

Alkalinity, meq/mL = [(0.86 mL)(0.2 meq/mL) - (1.002mL + 0.86 mL) ($10^{-3.42}$ - 0.0001)] / (1.002 mL)

= [(0.172 meq) - (1.862 mL) (0.00038019 meq/ mL- 0.0001 meq/mL)] / (1.002 mL)

= [(0.172 meq) - (1.862 mL) (0.00028019 meq/mL)] /(1.002 mL)

= [(0.172 meq) - (0.0005217 meq)] /(1.002 mL)

= [(0.17148 meq)] /(1.002 mL)

= 0.1711 meq/mL

Procedure for Polymer Concentration Determination
of
Water soluble amides

Equipment

50-mL Volumetric Flasks
Spec. 20 set at 610nm
Timer
Repeater Pipetor
5μ Filter paper
Suction filter apparatus

Solutions Needed

Acetate Buffer with Aluminum Sulfate
Bromine water
Starch-Cadmium Iodide Reagent
Sodium Formate

Solution Preparation

Acetate Buffer
Dissolve 82 g of Sodium Acetate, 60mL Acetic Acid, and 0.75g of Hydrated Aluminum Sulfate in deionized water and dilute to 1 liter. Adjust pH to 5.5 with Sodium Hydroxide.

Starch Indicator
Boil 11g of Cadmium Iodide in approximately 300mL deionized water for 15 minutes.
Dilute to approximately 800mL, bring to a boil again then add 2.5g of starch while stirring. Continue to heat and stir for 5 minutes. Filter the hot solution through 5μ filter paper with suction. Cool and dilute to 1 liter.

Bromine Water
Add enough Bromine to water to have excess Bromine at the bottom of the bottle.

Sodium Formate Solution
Dilute 1g of sodium formate to 100mL with deionized water.

Calibration Curve
Prepare a stock solution of polymer. Make dilutions of the polymer of 0-300ppm for the curve
Prepare a blank by adding 1mL of deionized water instead of polymer solution.
Run the polymer concentrations and blank as the samples.

Procedure:
To a 50 mL volumetric flask, add 5mL of buffer solution and 1mL of polymer solution.
Dilute to approximately 35mL with deionized water and add 1mL Bromine water. Swirl to mix.
Allow the mixture to react for 5 minutes.
Add 5mL of sodium formate solution.
After exactly 2 minutes add 5mL of the starch indicator solution.
Dilute to volume with deionized water.
Allow 10 minutes for color development.
Measure absorbance at 610nm.
Zero the Spectrophotometer with the reagent blank.

Note: All reagents should be American Chemical Society reagent grade.

APPENDIX 3

PHASE BEHAVIOR OF 2% CJ-2 SURFACTANT

Phase behavior tests were conducted with Karamay treated, diluted oil and Karamay stock tank oil. The samples with treated, diluted oil were prepared with 2% active CJ-2 surfactant and a series of Na_2CO_3 concentrations. Karamay brine was used for solution preparation. Phase tubes contained 3 mL oil and 6 mL aqueous.

The results from the diluted, treated oil samples were:

Na_2CO_3, %	OBSERVATIONS
1.4	Under optimum
1.6	Brown emulsion **(Near Optimum)**
1.8	Brown, stable emulsion **(Optimum)**
2.0	Brown, stable emulsion **(Slightly over optimum)**
2.2	Over optimum
2.4	Over optimum
2.6	Over optimum
2.8	Over optimum
3.0	Over optimum, lower aqueous becoming clearer
3.2	Over optimum, clear lower aqueous

The experiments conducted with Karamay stock tank oil were not nearly so definitive:

Na_2CO_3, %	OBSERVATIONS
0.5	Under optimum
1.0	Under optimum-some brown color and large oil droplets
1.2	Under optimum-some brown color and large oil droplets
1.4	Small amount of brown emulsion coats glass, but black bulk phase
1.6	Small amount of brown emulsion coats glass, but black bulk phase
1.8	Small amount of brown emulsion coats glass, but black bulk phase
2.0	Small amount of brown emulsion coats glass, but black bulk phase
2.4	The first time this mixture was shaken, it appeared uniform brown in color, but appears as gray on subsequent agitation

The optimal carbonate concentration range of stock tank is reduced slightly by adding lower molecular weight hydrocarbons to replace light ends that are lost after production. The stock tank oil continues to lose light ends when exposed to the atmosphere.

APPENDIX 4

RELATIVE PERMEABILITY CURVES AND MOBILITY CONTROL

Calculation Methods

Adequate mobility control during miscible oil recovery processes such as tertiary oil recovery using surfactants has been described and discussed in the literature. Methods to design adequate mobility control for micellar solutions were given by Gogerty *et al.*.[1] They discussed the use of relative-permeability curves to determine mobility requirements and methods to select representative relative-permeability curves from field core data. Trushenski *et al.*[2] and Chang *et al.*[3] (1978) described determination of mobility control requirements for surfactant/polymer corefloods from laboratory pressure measurements. The following discussion summarizes the discussions found in these papers to determine design criteria for adequate mobility control and methods to determine the required data for systems of interest and provides definition of appropriate terms used to discuss mobility control requirements.

Relative mobility of a fluid (λ) is defined as the relative permeability of that fluid divided by its apparent viscosity in the porous medium (k_r/μ). In two-phase flow, the total relative mobility (λ_t) is the sum of oil and water relative mobilities.

$$\lambda_t = \lambda_{rw} + \lambda_{ro} \tag{1}$$

Successful miscible flooding results in the formation of a stable oil bank in front of the miscible zone. Since both oil and water are displaced in a miscible flood, the combined flow of water and oil in the bank establishes the mobility requirements for the displacing fluids. Flow in the bank is governed by the principles of fractional flow for immiscible systems.[4] Total flow within the water-oil bank, neglecting capillary pressure, is given by:

$$q_t = \frac{k_e A \, \Delta P}{\mu_e L} = k A \left(\frac{k_{rw}}{\mu_w} + \frac{k_{ro}}{\mu_o} \right) \frac{\Delta P}{L} \tag{2}$$

where:

---

[1]Gogerty, W. B., H. P. Meabon, and H. W. Milton, Jr. *Mobility Control Design for Miscible-Type Waterfloods Using Micellar Solutions*. J. Pet. Tech., Feb. 1970, pp. 141-147.

[2]Trushenski, S. P., D. L. Dauben, and D. R. Parrish. *Micellar Flooding – Fluid Propagation, Interaction, and Mobility*. Soc. Pet. Eng. J., Dec. 1974, pp. 633-642.

[3]Chang H. L., H. M. Rikabi, W. H. Pusch. *Determination of Oil/Water Bank Mobility in Micellar-Polymer Flooding*. J. Pet. Tech., July 1978, pp. 1055-1060.

[4]Buckley, S. E. and M. C Leverett. *Mechanism of Fluid Displacementt in Sands*. Trans. of AIME, Vol.146 1942, pp. 107-116.

q_t = total fluid flow, cm^3/sec
A = area, cm^2
$\Delta P/L$ = pressure gradient, atm./cm
k_e = effective permeability, Darcy
k = base permeability for relative permeability measurements (k_w at 100% water saturation)
μ_e = effective viscosity, cP
$_w$ and $_o$ refer to water and oil respectively

Total relative mobility for a rock type is calculated as a function of fluid saturation using viscosities of the water and oil of interest and the relative permeability curves for that rock. The minimum mobility requirement or design mobility is shown by the minimum in the curve. The actual mobility of an oil bank mobilized by the chemical system may be equal to or higher than the minimum mobility, but the bank does not have a mobility less than the minimum. Mobility buffers, therefore should have a mobility value less than or equal to the design mobility over the range of expected fluid velocities to be found in the reservoir.

In the reservoir, flow rates of injected fluids are high around the wellbore and decline as fluids flow in a radial pattern toward producers. The flow properties of mobility-control chemicals (polymers) are dependent on shear rate experienced by the fluids. Therefore, their mobility characteristics will depend on flow rate through porous media. In the laboratory, these properties are measured by pumping mobility control solutions through core of interest and measuring pressure differentials as a function of fluid flow rate. This mode of measurement will take into account pore structure of the porous media and its effect on fluid movement as well as non-Newtonian behavior (effect of shear rate) of the polymer.

The relative mobility of the buffer solution is calculated from the flow measurement data using equation 3. The mobility is then compared with the design mobility to determine if the chemical formulation exhibits adequate mobility control characteristics for the experimental conditions under study.

$$\lambda_t = \frac{k_e}{k\,\mu_e} = \frac{q\ L}{kA\,\Delta P} \tag{3}$$

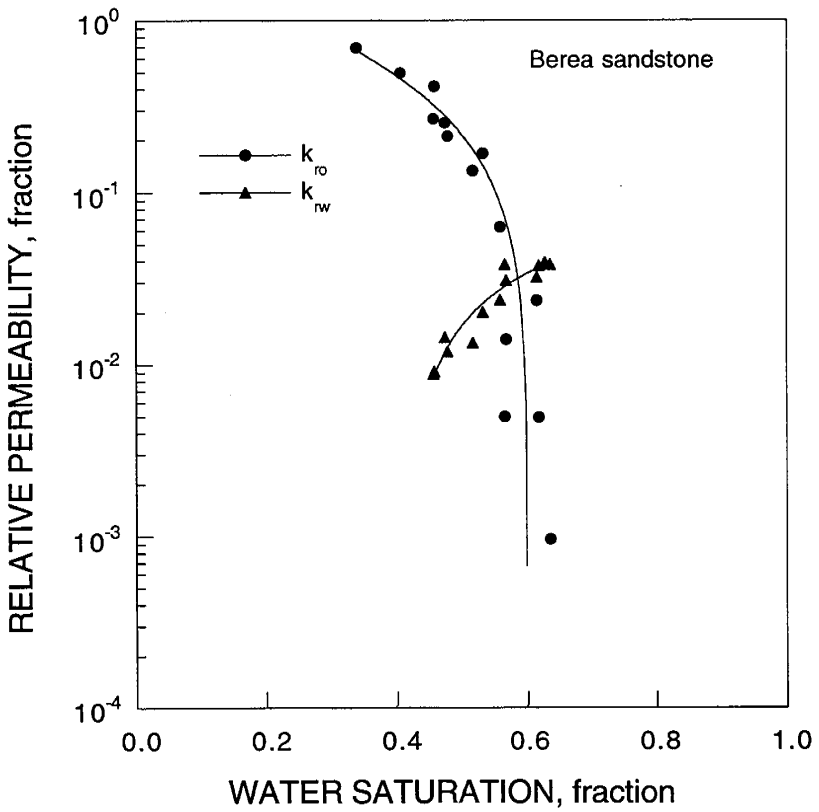
Mobility Calculations for the Karamay Reservoir ASP Laboratory Study

Berea Sandstone Core

Figure A5-1 shows a relative permeability curve for the Berea core used in the Karamay laboratory coreflood evaluation study. Figure A5-2 shows total mobility calculations (equation 1) for 17 and 25 cP oil based on the Berea sandstone relative permeability curves.

Polymer mobility during coreflood experiments should be less than the minimum total mobility values (≈0.02 to 0.025 cP^{-1}) for the Karamay oil used in the study.

A series of polyacrylamide polymer solutions of different concentrations were pumped through Berea sandstone core to determine mobility as calculated from equation 3 as a function of flow rate (frontal velocity) of the fluid. Figure A5-3 shows results for the MO3000H-SF polymer. Polymer solutions that contained no added salt showed adequate mobility control characteristics for all concentrations tested. When salt (Na_2CO_3) was added, however, measured mobility was marginal compared to the calculated mobility requirements.



FigureA5-1 Steady-state Relative Permeability Curves for Berea Sandstone with Average Permeability to Brine of Approximately 200 mD

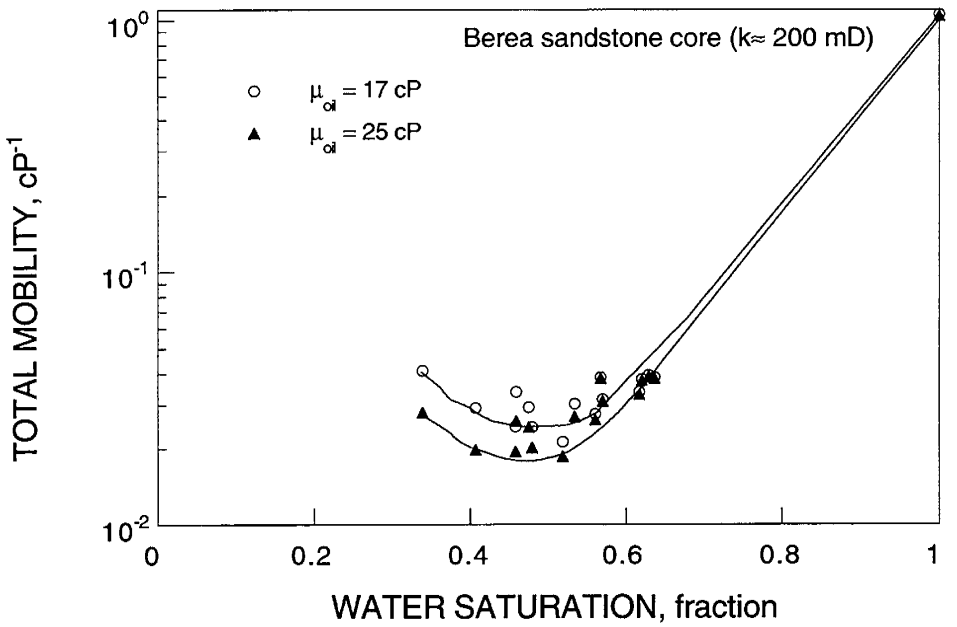


Figure A5-2 Total Mobility as a Function of Water Saturation for Different Viscosity Oils

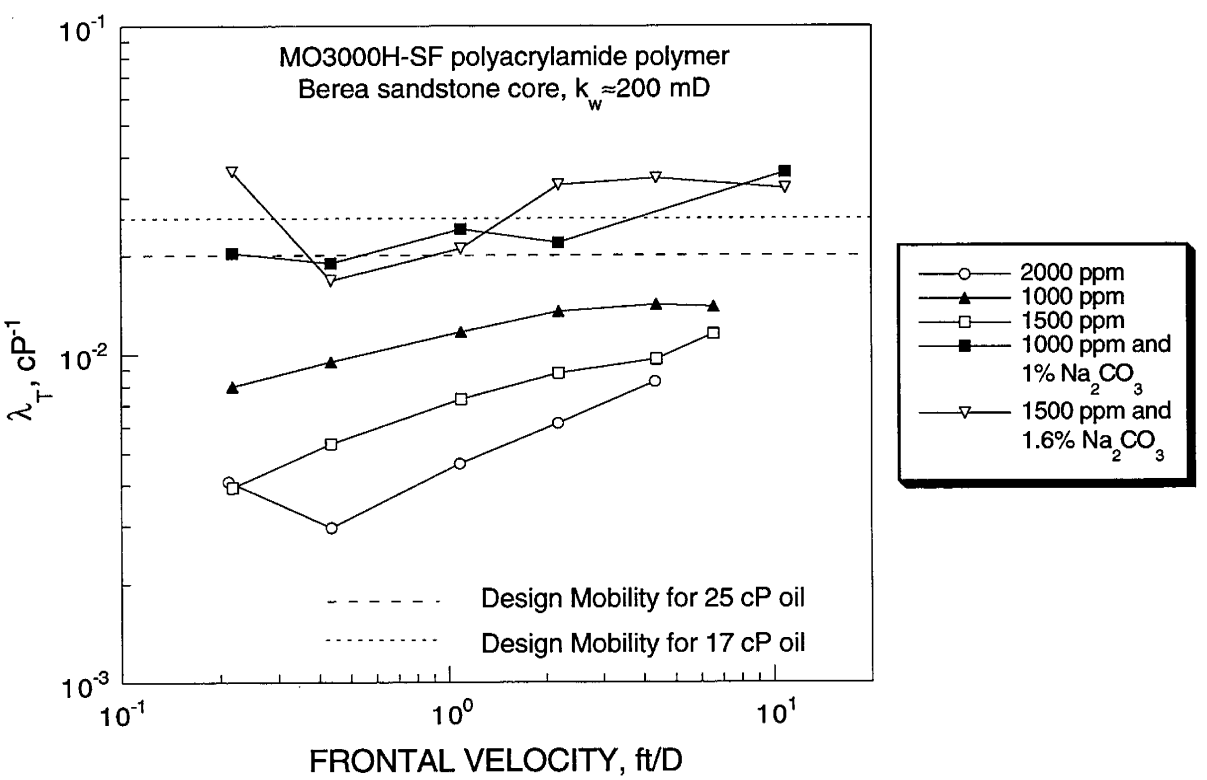


Figure A5-3 Total Mobility as a Function of Frontal Velocity of Polymer Solutions

Polymer concentration selected for the oil recovery tests was 1500 ppm. In the Berea sandstone tests, some experiments were conducted with polymer prepared in Karamay river water and some with an additional 0.5 to 1% NaCl added to the polymer mobility slug. Table A5-1 shows the maximum total mobility as calculated from equation 3 for the different corefloods conducted in Berea sandstone.

Mobility control during most tests appeared to be adequate. Total mobility during KMAY-6 and KMAY-7, however, are very close to the required design mobility. KMAY-5, KMAY-6, and KMAY-7 were conducted using the same amount of surfactant (2% CJ-2). Recovery efficiency for KMAY-5 was 93%. For the tests with poorer mobility control (KMAY-6 and KMAY-7), recovery efficiency was 78% and 71%.

Even small variations in mobility control can affect oil production under laboratory. Extra care should therefore be taken to ensure that adequate mobility control is maintained in the field ASP project.

Table A5-1 Calculated Total Mobility During Chemical Flood for Tests Conducted in Berea Sandstone

Core I. D.	λ_t, cP^{-1}	Flood Type
KCF-1	0.00053	ASP
KCF-2	0.0018	A
KCF-3	0.0032	AP
KCF-4	0.0026	ASP
KCF-6	0.0018	ASP-higher A
KMAY-1	0.0035	ASP
KMAY-2	0.0040	ASP
KMAY-3	0.0017	ASP-higher A
KMAY-4	0.0062	ASP
KMAY-5	0.0067	ASP
KMAY-6	0.018	ASP
KMAY-7	0.021	ASP
Radial Core	0.0055	ASP

Karamay Reservoir Core

Figure A5-4 shows total mobility calculated for Karamay reservoir core, Y2-1, steady state relative permeability data using an oil viscosity of 17.7 cP and water viscosity of 1.03 cP. The minimum mobility based on this relative permeability curve for the Karamay reservoir is approximately 0.03 cP^{-1}.

For the oil recovery experiment using S-42-2, adequate mobility control was achieved as λ_t = 0.003 cP^{-1} was calculated from the pressure data. For other tests using reservoir core, pressures generated during injection of aqueous fluids continued to increase. A reliable mobility value can not be obtained. Possible problems with clay swelling or retention of polymer could be the cause of injection problems. Low oil recovery may be a result of rock quality and rock heterogeneity rather than poor mobility control. However, for good quality rock, it appears that 1500 ppm polyacrylamide polymer in 0.5% NaCl provides adequate mobility control.

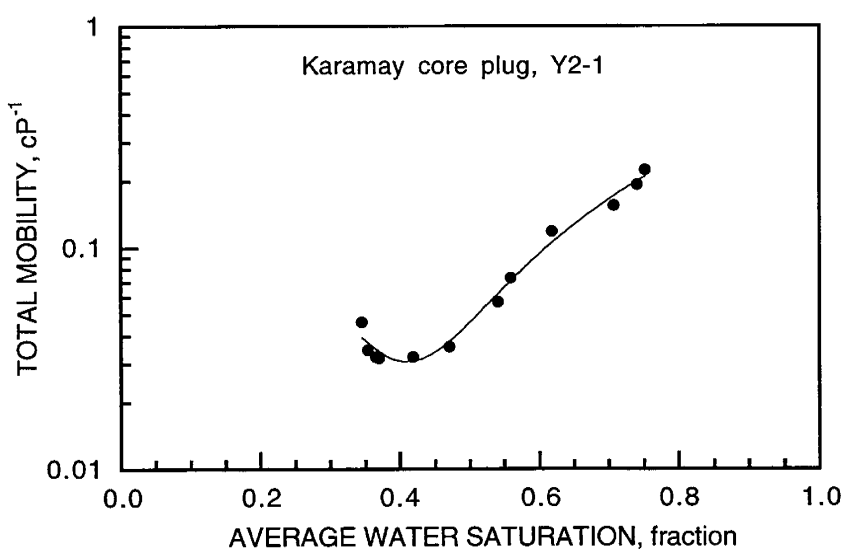


Figure A5-4 Design Mobility Calculated from Steady-State Relative Permeability Curve for Karamay Core Plug, Y2-1