EVALUATION OF POLYMERIC DAMAGE BASED UPON IMPROVED FLOWBACK ANALYSIS

Michelle C. Flippen and B. Helena Yang
BJ Services Company

Abstract

The removal of polymers utilized in oilfield applications is important to the conductivity and productivity of a well. Unbroken gel residue and dynamically formed filter cake on the formation faces are two forms of damage resulting from drilling, fracturing, gravel packing and workover operations.

Monitoring the extent of polymeric damage and its cleanup through removal treatments is best achieved through the analysis of flowback samples. This form of analysis can provide valuable information regarding polymer degradation downhole and be used as a quantitative profile for the amount of treatment load recovered. Flowback samples can be tested before and after treatments to determine the total carbohydrate content, which is a measurement of sugar concentration, in pounds per thousand gallons. Guar, cellulose, starch, xanthan and other polysaccharides used as viscosifying agents are examples of complex sugars. Although high carbohydrate levels are a symptom of damaged wells, it is misleading to conclude that lower carbohydrate content equates to a lesser degree of damage. Other factors, such as bacterial presence, breaker activity and size distribution of polymer fragments, contribute significantly to the results of a flowback analysis.

This paper presents an improved method to effectively analyze flowback samples. Laboratory protocols are provided and include tests for carbohydrate content, molecular weight distribution, enzyme/bacteria detection and viscosity measurements. This improved flowback analysis provides a method to evaluate polymer load recovery and to detect any polymer damage downhole. Several field studies are also included to demonstrate this comprehensive analytical procedure and how it supplies a more conclusive post-treatment evaluation.

Introduction

Natural, water soluble polymers have a long history of use in the oil and gas industry due to their unique fluid rheology characteristics, proppant carrying ability and high temperature stability. Applications include drilling, fracturing, gravel packing, enhanced recovery, completion and workover operations. However, these polymers can leave behind unbroken gel filter cake on the formation faces or insoluble residues within the proppant pack. At times the concentration of the filter cake becomes so high that breaker additives are unable to thoroughly degrade it. Insoluble residues, high molecular weight fragments and polymer degradation products are no longer soluble and fall out of solution. These degradation products can settle within the proppant pack and impair permeability. Since the damage produced by natural polymers can have a negative effect on well productivity, it is important to ensure that most of the polymer is returned after a treatment.[1-4]

Flowback waters have previously been used as a source of information regarding load recoveries following

fracturing treatments. One method of quantifying cleanup was to measure chlorides, sulfates and/or specific gravity of the flowback and compare it with properties of the formation water. However, the use of chlorides, sulfates, and/or specific gravity serves only as a measure of the water load recovered. This type of test gives no information about the amount of polymer returned, or more important, the polymer left downhole. An alternative method was to monitor the viscosity of the flowback samples over time. Reduced viscosity observed in return flow fluids was considered proof of optimum gel degradation. A problem arises when the formation's naturally produced water dilutes flowback samples, leading to a misinterpretation of adequately broken gels. Recently, flowback waters have been recognized as a method of evaluating polymer load recovery through the testing for polymer content in flowback samples. Pope reported that a more quantifiable approach to describing fracture cleanup is performed by determining the amount of guar returned from the fracture during flowback.[5] Tyssee/Vetter also used analysis of return waters to support arguments made by their study regarding water-soluble polymers.[6]

This paper presents an improved method to effectively analyze flowback samples for polymeric damage. Analysis can take place before and after treatments to help determine the extent of polymer damage produced by guar, cellulose, starch, xanthan and other natural polymers. Samples are tested for total carbohydrate content (TCC), molecular weight distributions, enzyme/bacteria detection, as well as the standard pH and viscosity measurements. Several of these procedures have been applied previously to the testing of polymer and breaker system chemistries. Tyssee and Vetter introduced the concept of TCC in the early 1980's. In their paper correlations were made between the total organic content and carbohydrate content of return waters as a function of residence time under simulated reservoir conditions.[6] Testing broken fluids for the molecular weight distribution has also been used before to demonstrate the damage caused by high molecular weight polymer fragments or residues. Gall and Raible used size exclusion chromatography (SEC) to monitor the reduction in molecular size of broken polymer solutions. These results were then correlated with solution viscosity to conclude that low solution viscosity did not guarantee reduction of polymer molecules to non-damaging sizes.[7] Volk *et al.* used high pressure liquid chromatography (HPLC) techniques to determine the presence and molecular weight distribution of fracture fluid polymers passing through cores.[8] This improved flowback analysis emphasizes the roles that carbohydrate content and molecular weight distributions play in determining the extent of polymer retained within the formation. It has been found that this technique can identify damaged wells and direct further treatment to improve permeability and conductivity, thus increasing production.

Flowback Analysis Theory

Carbohydrate Analysis. Carbohydrates are a wide variety of polyhydroxylated aldehydes and ketones commonly called sugars.[9] Carbohydrates can be classified on the basis of their hydrolysis to simple sugars. Simple sugars, or monosaccharides, are compounds like glucose and fructose that cannot be hydrolyzed into smaller molecules. For example, sucrose (table sugar) is a disaccharide (two sugars) which is made up of one glucose molecule linked to one fructose molecule. Polysaccharides are carbohydrates in which tens, hundreds, even thousands of simple sugars are bonded together through specific linkages. Guar, cellulose and starch are the three most widely used polysaccharides in the oil and gas industry. Studies have shown that guar consists of a mannose backbone with galactose side chain bonded to every other mannose unit.[10]

Flowback samples from damaged wells contain mixtures of mono-, di-, or polysaccharides segments. TCC in pounds per thousand gallons (lb/Mgal) is a measurement of the total concentration of monosaccharides or sugar units in the sample. For instance, 40 lb/Mgal of table sugar and 40 lb/Mgal of guar polymer will give corresponding carbohydrate concentrations. TCC is measured by a procedure referred to as an anthrone test and is based on the principle that carbohydrates are dehydrated when reacted with strong mineral acids under nonoxidizing conditions.[11] Hydrolysis, or dehydration of polysaccharides breaks them down into their individual monosaccharide units, which are then converted to furfural or hydroxymethylfurfural by concentrated sulfuric acid. These cyclic aldehydes, in turn, will react with anthrone to form a mixture of colored condensation products, and is the basis of the anthrone test for carbohydrates.

Colorimetric methods are utilized to quantitate the carbohydrate concentration because of its simple means for determining minute quantities of a substance. Previous studies have demonstrated the use of anthrone in qualitative and quantitative tests for various carbohydrates and their derivatives.[12-14]

Molecular Weight and Viscosity. Molecular weight refers to the effective size occupied by a polymer chain in solution.[15] Solution viscosity has an exponential relationship with a polymer's molecular weight and as a result, small reductions in polymer size produce substantial decreases in viscosity.[7] Brannon and Tjon-Joe-Pin emphasized this point by characterizing breaker systems based upon molecular weight distributions of broken gel solutions over an extended test period. It was concluded that fluids with a viscosity of less than 5.0 cps still contained polymer fragments exceeding 1,200,000.[16] Yet, for years Darcy's equation has led many to interpret the low viscosity of returned fluids as evidence of high permeabilities and conductivities downhole. The effect of large molecular weight fragments and its restricted flow through a formation were not taken into account. Therefore, it would be misleading to conclude that broken gel viscosity equates to polymer degradation of non-damaging proportions.

Although SEC and HPLC are valuable tools for determining molecular weight distributions, these procedures are not cost or time effective for the testing of multiple flowback samples. In this analysis molecular weight distributions were determined using an Ultra-Filtration Molecular Weight Cut-off (MWCO) technique. This technique involves separating the variously sized polymer fragments in broken gel solutions across a semi-permeable membrane using centrifugation. These membranes are capable of separating fragments to 1,200, 300, 100, 30, 10 and 5 thousand nominal molecular weight limits (NMWL). A previous study by Brannon and Tjon-Joe-Pin verified this method for accuracy by CHNOS. CHNOS analysis may be used for quantitative determination of carbon, hydrogen, nitrogen, sulfur and oxygen content present in a wide range of organic and inorganic samples. Samples of filtrate from the MWCO test were subjected to a flash combustion at 1200°C in a reactor which converts organic and inorganic substances into combustion products. The resulting combustion gases were passed first through a separation column and then a thermal conductivity detector which transmits a response signal proportional to the concentration of the elements in the sample. The results indicated that the carbon content was consistent with the molecular weight distributions and had not been altered by water weight.[17]

Additional Factors. Many other factors must be taken into account when using this technique to assess polymer damage. First, breaker activity or the presence of sugar reducing bacteria in the flowback sample

can indicate that polymer degradation is still occurring. This could influence the results of the anthrone test, molecular weight distribution and viscosity measurements and lead to the conclusion of insignificant or minimal polymer damage. The formation's ability to produce water also must be considered when applying this procedure to the assessment of polymer damage. Flowback samples from water producing wells can be diluted, giving lower carbohydrate and viscosity values. These examples show a few reasons for reduced carbohydrate content in which polymer damage could exist.

Laboratory Procedures

Flowback Sampling. Flowback samples were sent in for low performance wells to test for possible polymeric damage. After fracturing or polymer removal treatments samples were collected at scheduled time intervals, for as long as possible, to evaluate cumulative polymer load recovery. The volume of fluid returned, in barrels, and the amount of fluid per hour were recorded for each sample.

Samples were recommended to be refrigerated or preserved by a biocide. This prevents bacteria from growing and consuming any contained sugars, which can cause inaccuracy of the laboratory test results.

Anthrone Analysis. An anthrone analysis was performed by adding a 2% anthrone-sulfuric acid solution into diluted flowback samples. The percent transmittance (%T) of the developing color was then determined using a spectrophotometer at a wavelength of 625nm. A standard curve was developed by measuring the %T of several known carbohydrate concentrations in fluid condition similar to that of the flowback samples. The curve was then plotted using the log of the measured %T as the ordinate and the carbohydrate concentration as the abscissa. The TCC of the flowback samples was then extrapolated from the standard curve. The margin of error for this procedure has been observed to be $\pm$2%.

Molecular Weight Distribution. The molecular weight distributions were evaluated using the ultra-filtration MWCO technique. This analysis provides a measure of the weight percentage of soluble and insoluble material present in various size ranges. The filtration membranes utilized were Ultra-Free CL, low binding cellulose filters by Millipore and are capable of separating polymer fragments to 1,200, 300, 100, 30, 10, and 5 thousand nominal molecular weight limits (NMWL). Calibration of the membranes was based on performance characteristics for the retention or passage of single solute marker solutions of proteins or dextrans.

Flowback samples with any inorganic solids or fines were first passed through a one micron syringe tip filter. This prevents plugging of the membranes which could shift the molecular weight distribution. Each molecular weight cut-off tube was weighed before the addition of the sample. One milliliter of the flowback sample was then added to the tube and centrifuged at 2500Gs for 30 minutes. The weight of the tube was again measured after centrifugation to arrive at the amount of sample that had passed through the membrane. A margin of $\pm$6% error was observed for the MWCO analysis based on reproducibility.

Viscosity Measurements. Viscosity readings were conducted at ambient temperature using Fann 35A Viscometer. The measurements were taken with an R1:B1 rotor:bob configuration at 300 rpm, which provides fluid viscosity at 511s^{-1}. This is the standard procedure for the evaluation of broken gel viscosity.

Bacteria Detection. The presence of bacteria was determined visually using a microscope at approximately 1000X magnification. An estimated bacterial cell count is given in cells per milliliter. These numbers were established by using a counting chamber or slide with an etched grid consisting of one square that is divided into 400 smaller squares.

Enzyme Breaker Detection. The detection of enzyme breaker activity was made using a Folin-Ciocalteu reagent spot test.[18] Two drops of the reagent were added to about 10 mL of flowback sample and agitated vigorously and set aside for 30 minutes. The appearance of a dark blue color indicates the presence of enzymes, with a detection limit of 1.25 mg/L.

Field Trials and Results

A field study of several wells was conducted to evaluate polymeric damage and/or polymer load recovery using this improved flowback analysis. TCC, molecular weight distribution, pH, viscosity and enzyme/bacteria detection were performed on all flowback samples received. The cumulative carbohydrates are presented to provide a quantitative profile for the amount of treatment load recovered. Based upon our database, fractured wells which are producing as expected have demonstrated at least 80% of expected load recovery within four months. These wells typically have been found to have a TCC in the range of 0.1 to 0.4 lb/Mgal. The viscosity of all samples was observed to be less than 5.0 cps, suggesting that even wells whose viscosity measurements are low could still have tremendous polymeric damage.

Well A: Alberta, Canada. A study was conducted on a Rock Creek sandstone formation with a bottomhole static temperature (BHST) of 127°F. In 1991 this well was fractured with a crosslinked methanol system, and production was observed to be low compared to off-set wells. A flowback sample was analyzed to test for possible polymeric damage and was found to have a TCC of 10.3 lb/Mgal, an average molecular weight ($AvMW_{1,200K}$) of 122K, 4% of the fragments greater than 1.2 M and 32.1% is greater than 100K. These results indicated there was polymeric damage downhole even after having been in production for five years.

A polymer removal treatment was performed on this well and flowback samples were collected and analyzed afterwards to monitor the treatment results. The molecular weight distributions of a few samples are illustrated in Fig. 1. TCC, $AvMW_{1,200K}$, pH, viscosity and bacteria detection data are shown in Table 1. The first few samples after the removal treatment were observed to have 50-60 lb/Mgal of TCC, $AvMW_{1,200K}$ of 531-822K and weight percent above 1.2 M was 67%. The increase of TCC, $AvMW_{1,200K}$ and the molecular weight distribution indicated that the well was cleaning up.

Two months following the treatment, the TCC level of the sample had declined to 12.0 lb/Mgal and $AvMW_{1200K}$ had decreased from 822K to 75K. This indicates that polymers were still flowing back as a result of the polymer removal treatment. Meanwhile, the production had increased from 1 MMCFD to 1.5 MMCFD, a 50% overall increase.

Well B: South Texas. Well B is a Wolf Camp sandstone formation with a BHST of 160°F and a depth interval of 11,660-11,800 ft. This well was fraced with a guar-borate-oxidizer fluid system and resulting well performance was much lower than expected, with less than 10% of the frac load being returned over

a month period. A flowback sample was collected and evaluated and found to have a TCC of 20 lb/Mgal, $AvMW_{1,200K}$ of 903K and 68% of the fragments larger than 1.2 M.

As a result of this analysis, a polymer damage removal treatment was performed and post-treatment samples were evaluated. The $AvMW_{1,200K}$, percent above 1.2 M, TCC numbers, pH and viscosity measurements are illustrated in Table 2. The molecular weight distribution of the flowback samples are shown in Fig. 2.

The high TCCs returned in earlier flowback samples are characteristic of a successful removal treatment. Often, a five- to 10-fold increase in the TTC is observed right after the treatment, which suggests polymer filter-cake degradation. Also the cumulative polymer load return was calculated and presented in Table 3.

Well C: Hutchinson, Texas. Well C is a Cleveland sandstone formation with a BHST of 145°F. This well was fractured with a 30 lb/Mgal of monoborate, crosslinked guar fluid with sodium persulfate and a delayed release oxidizer breaker system. This well was also producing much less than expected with minimal load return over a six-month period. Two flowback samples were analyzed to evaluate for possible polymer damage, and the results suggested a tremendous amount of polymer load was yet to be recovered. A polymer removal treatment was performed six months after the fracturing treatment, with a shut-in of one week.

The TCC, $AvMW_{1,200K}$, weight percent greater than 1.2 M, pH and viscosity are illustrated in Table 4. The molecular weight distributions of the samples obtained from this well are shown in Fig. 3. The TCC was observed to increase compared to the numbers obtained after the fracturing treatment, indicating that more polymer load was recovered from this well. The $AvMW_{1,200K}$, weight percent above 1.2 M and the molecular weight distribution also indicate that the unbroken gel downhole was degrading to smaller fragments and flowing back to the surface.

Immediately after the removal treatment, this well kicked-off with a three-fold increase in production from 50 MCFD to 152 MCFD. Over the next six days, production stabilized at 290 MCFD, for an overall six-fold increase.

Well D: Southeast Texas. A case study was conducted on a dry gas well fractured using 1,600 bbls of 40 lb/Mgal CMHPG-zirconium-crosslinked fracturing fluid with a guar-linkage-specific enzyme (GLSE) breaker. This well is a Lobo/Wilcox sandstone formation with a BHST temperature of 292°F. Following the frac treatment, the well was shut in for one day and afterwards had a flowback rate of 3 bbl/min.

In Table 5 the $AvMW_{1,200K}$, weight percent greater than 1.2 M, pH, viscosity, enzyme and bacteria detection data are presented for 9 of 32 flowback samples tested from this well. The molecular weight distributions for these nine samples are illustrated in Fig. 4. The decreasing trend of the molecular weight distribution, $AvMW_{1,200K}$ and weight percentage above 1.2 M indicates that the polymer downhole was gradually degrading to the smaller sized fragments as more fluid was returned.

The load recovery data is presented in Table 6 and includes 16 of 32 data points collected. The cumulative polymer recovery was calculated according to the fluid return level in barrels and TCC of each flowback

sample. At 189 bbls and 616 bbls, the polymer load returned was calculated to be 282 and 1,100 lbs, respectively. The total amount of polymer pumped on this frac job was 2,688 lbs. At 710 bbls of flowback, the expected polymer load returned would be 1,198 lbs. A polymer load of 1,204 lbs of polymer was calculated to have returned, which would suggest 100% recovery up to this interval.

Well E: North Texas. Flowback samples were analyzed for a Barnett shale formation that is highly fractured and typically retains 70% of the frac load. The perforated interval was 6,765-6,959 ft. with a BHST of 200°F. The well was stimulated with a guar-borate-crosslinked system containing a delayed-release persulfate and ammonium persulfate as gel breakers. The $AvMW_{1,200K}$, weight percentage above 1.2 M, TCC, pH and bacteria detection results are presented in Table 7. The molecular weight distributions for the flowback samples are illustrated in Fig. 5, with 69-80% of the polymer fragments in the greater than 1.2 M range. In addition, the $AvMW_{1,200K}$ was in the range of 854K to 973K with 69-80% above 1.2 M. This data represents a typical distribution profile of a fracturing treatment broken with oxidizers.

Well F: Rocky Mountains, Colorado. A flowback sample was analyzed from a Frontier sandstone well, having BHST of 265°F. This well was fractured with guar-zirconium fluid, utilizing controlled-release conventional enzyme breaker. As shown in Fig. 6, the molecular weight distribution after fracturing treatment is towards the 1.2 million range and TCC was calculated to be 33 lb/Mgal. Although the viscosity of the fluid was only 5.2 cps, there was a high concentration of large polymer fragments detected in the flowback sample. Since production has been much lower than expected and the flowback sample was analyzed six months after treatment, this well would be an excellent candidate for a polymer damage removal treatment.

Conclusions

1. Old methods of analysis for water load recovery are not sufficient for assessing polymer damage or evaluating polymer load recovery.
2. This paper presents test procedures including total carbohydrate content, molecular weight distribution, viscosity measurement, enzyme and bacteria detection.
3. This improved flowback provides a more conclusive test for the evaluation of polymer damage and a method to calculate polymer load recovery.
4. Laboratory procedures have been successfully applied to several field studies and a promising assessment of polymeric damage has been shown.
5. A biocide system is to be developed and studied in order to effectively preserve the flowback samples.

Acknowledgment

The authors wish to thank the management of BJ Services for their support and permission to publish this paper. We would also like to thank Robert Tjon-Joe-Pin, whose contributions and guidance made this paper possible. Special thanks go to Doris Porter for preparing the manuscript.

References

1. Roodhart, L.P., Kuiper, T.O.H. and Davies, D.R.: "Proppant Pack Impairment During Hydraulic Fracturing," paper SPE 15629 presented at the 61st SPE Annual Technical Conference, New Orleans, Oct. 5-8, 1986.
2. Almond, S.W.: "Factors Affecting Gelling Agent Residue Under Low Temperature Conditions," paper SPE 10658 presented at the 1982 SPE Formation Damage Control Symposium, Lafayette, Mar. 24-25.
3. Penny, G.S.: "An Evaluation of the Effects of Environmental Conditions and Fracturing Fluids Upon the Long-Term Conductivity of Proppants," paper SPE 16900 presented at the 62nd SPE Annual Technical Conference, Dallas, Sept. 27-30, 1987.
4. Brannon, H.D. and Tjon-Joe-Pin, R.M.: "Biotechnological Breakthrough Improves Performance of Moderate to High-Temperature Fracturing Applications," paper SPE 28513 presented at the SPE 69th Annual Technical Conference and Exhibition, New Orleans, Sept. 25-28, 1994.
5. Pope, D. *et al.*: "Field Study of Guar Removal from Hydraulic Fractures" paper SPE 31094 presented at the SPE International Symposium on Formation Damage Control, Lafayette, Feb. 14-15, 1995.
6. Tyssee, D.A. and Vetter, O. J.: "Chemical Characterization Problems of Water-Soluble Polymers," paper SPE 8977 presented at the SPE 5th International Oilfield and Geothermal Chemistry Symposium, Stanford, May 28-30, 1980.
7. Gall, B.L. and Raible, C.J.: "Molecular Size Studies of Degraded Fracturing Fluid Polymers," paper SPE 13566 presented at the International Symposium on Oilfield and Geothermal Chemistry, Phoenix, April 9-11, 1985.
8. Volk, L.J. *et al.*: "A Method for Evaluation of Formation Damage Due to Fracturing Fluids," paper SPE/DOE 11638 presented at 1983 SPE/DOE Symposium on Low Permeability, Denver, March 14-16.
9. McMurry, J.: *Organic Chemistry*, 3rd edition, Brooks/Cole Publishing Company, Pacific Grove, CA (1992) 916.
10. Chatterji, J. and Borchardt, J.K.: "Applications of Water-Soluble Polymers in the Oil Field", *JPT* (Nov. 1981) 2046-2048.
11. Strong, F.M. and Koch, G.H.: *Biochemistry Laboratory Manual*, Wm.C. Brown Company Publishers, Dubuque, Iowa (1981) 66.
12. Dreywood, R.: "Qualitative Test for Carbohydrate Material," *Ind. Eng. Chem. Anal.* Ed. 18, (1946) 499.
13. Morris, D.L.: "Quantitative Determination of Carbohydrates With Dreywood's Anthrone Reagent," *Science* **107** (1948) 254.
14. Viles, F. J., Silverman, L.: "Determination of Starch and Cellulose with Anthrone," *Anal. Chem.* **21** (1949) 950.
15. Seymour, R.B. and Carraher, C.E.: *Polymer Chemistry*, Marcel Dekker, Inc., New York City (1992) 82, 87.
16. Brannon, H.D. and Tjon-Joe-Pin, R.M.: "Characterization of Breaker Efficiency Based Upon Size Distribution of Polymeric Fragments Resulting from Degradation of Crosslinked Fracturing Fluids,"

paper SPE 36496 presented at the SPE Annual Technical Conference and Exhibition, Denver, Oct. 6-8, 1996.

17. Brannon, H.D. and Tjon-Joe-Pin, R.M.: "Characterization of Breaker Efficiency Based Upon Size Distribution of Polymeric Fragments," paper SPE 30492 presented at the SPE Annual Technical Conference and Exhibition, Dallas, Oct. 22-25, 1995.
18. Jungreis, E.: *Spot Test Analysis: Clinical, Environmental, Forensic, and Geochemical Applications,* Chem. Anal. Vol. 75, John Wiley & Sons, Inc. New York, NY (1985) 95.

SI Metric Conversion Factors

cp x 1.0*	E-03	= Pa's
°F (°F-32)/1.8		= °C
bbl x 1.589 893	E-01	= m^3
lb x 4.535 924	E-01	= kg
gal x 3.785412	E-03	= m^3

* Conversion factor is exact.

Table 1 - Molecular Weight, TCC, PH, Viscosity, and Bacteria Detection of Well A

ID#	$AvMW_{1,200K}$	Weight % > 1.2 M	TCC (lb/Mgal)	pH	Viscosity (cps)	Bacteria (cells/mL)
Before Removal Treatment						
1	122K	4%	10.3	6.58	1.8	$10-10^3$
After Removal Treatment						
2	822K	67%	61	6.73	6.8	10^8-10^{11}
4	531K	40%	46.0	7.09	5.0	$10-10^3$
6	268K	11%	33.5	7.30	4.8	10^5-10^8
8	259K	11%	26.0	7.39	3.1	10^3-10^5
10	164K	5%	14.8	6.73	2.3	$10-10^3$
11	64K	1%	12.5	6.74	2.0	$10-10^3$
12	73K	2%	13.5	6.79	2.8	$10-10^3$
14	75K	3%	12.0	6.99	1.2	10^5-10^8

Table 2 - Molecular Weight, TCC, Ph, and Viscosity of Well B

ID#	Time	$AvMW_{1,200K}$	Weight % > 1.2 M	TCC (lb/Mgal)	pH	Viscosity (cps)
After Fracturing Treatment						
I	1 month	903K	68%	20.0	--	3.3
After Removal Treatment						
1	30 min	9K	0%	180.0	7.44	2.5
2	65 min	14K	0%	140.0	7.36	2.5
5	130 min	111K	7%	29.0	7.44	2.8
9	212 min	128K	9%	22.5	7.34	2.8
10	233 min	185K	13%	28.0	7.24	2.8
13	292 min	41K	2%	25.0	7.14	3.0
16	29 hrs	32K	1%	26.0	7.07	2.9
17	53 hrs	18K	0%	37.5	6.99	2.7
19	101 hrs	13K	0%	31.5	7.02	2.9

Table 3 - Cumulative Polymer Load Recovery of Well B

ID #	Time	Fluid Level (bbls)	TCC (lb/Mgal)	Cumulative Recovery (Lbs)
1	30 min	102	180.0	771
2	65 min	194	140.0	1312
3	85 min	248	29.0	1378
4	112 min	337	27.0	1479
5	130 min	391	29.0	1545
6	146 min	439	36.5	1618
7	161 min	484	20.0	1656
8	188 min	564	19.0	1720
9	212 min	637	22.5	1789
10	233 min	700	28.0	1863
11	254 min	763	22.0	1921
12	275 min	826	20.5	1975
13	293 min	878	25.0	2030

Table 4 - Molecular Weight, TCC, Ph, and Viscosity of Well C

ID#	Time	$AvMW_{1,200K}$	weight % > 1.2 M	TCC (lb/Mgal)	pH	Viscosity (cps)
After Fracturing Treatment*						
I	2 months	717K	53%	10.5	5.70	1.2
II	4 months	648K	48%	6.9	6.34	1.2
After Removal Treatment						
1	168 hrs	42K	0%	20.0	--	--
2	171 hrs	68K	3%	33.5	6.35	1.4
3	173 hrs	53K	1%	36.5	6.55	1.4
4	175 hrs	12K	0%	48.5	6.28	1.2
5	176 hrs	48K	3%	40.0	6.42	1.2
6	192 hrs	12K	0%	47.0	6.44	1.2
7	194 hrs	12K	0%	47.0	6.29	1.2
8	197 hrs	15K	0%	38.0	6.16	1.2

*The removal treatment was started six months after the fracturing treatment, with a shut-in of one week.

Table 5 - Molecular Weight, PH, Viscosity, and Enzyme/Bacteria Detection of Well D

ID#	$AvMW_{1,200K}$	Weight % > 1.2 M	pH	Viscosity (cps)	Enzyme Detection	Bacteria (cells/mL)
1	955K	79%	7.76	21.6	+	10^{8}-10^{11}
6	816K	67%	7.00	2.4	+	> 10^{11}
8	561K	35%	7.62	4.0	+	10^{5}-10^{8}
12	346K	14%	7.53	3.2	+	10^{3}-10^{6}
21	195K	1%	7.56	3.0	+	10^{3}-10^{5}
23	161K	0%	7.40	--	+	10-10^{3}
27	149K	2%	6.86	2.4	+	10-10^{3}
28	120K	2%	6.68	2.5	+	10^{5}-10^{8}
30	48K	2%	6.73	--	+	10^{3}-10^{5}

Table 6 - Cumulative Polymer Load Recovery for Well D

ID #	Fluid Level (bbls)	TCC (lb/Mgal)	Cumulative Recovery (lbs)
1	15	39.5	25
3	42	40.8	73
5	61	41.8	107
7	107	45.0	190
9	147	21.7	250
11	189	16.5	282
13	252	52.5	417
15	312	46.0	532
17	383	45.5	665
19	436	49.5	773
21	507	49.5	928
23	581	38.0	1055
25	616	31.5	1100
27	640	32.5	1133
29	665	26.5	1163
32	710	19.0	1204

Table 7 - Molecular Weight, TCC, PH, and Bacteria Detection of Well E

ID#	$AvMW_{1,200K}$	Weight % > 1.2 M	TCC (lb/Mgal)	pH	Bacteria (cells/:nL)
1	973K	81%	24.3	8.56	10^{8}-10^{11}
2	920K	75%	28.0	7.72	10^{3}-10^{5}
3	924K	76%	26.1	7.59	10-10^{3}
4	885K	72%	25.8	7.65	10^{5}-10^{8}
5	899K	74%	26.1	7.45	10^{3}-10^{5}
6	897K	74%	24.2	7.52	10^{5}-10^{8}
7	920K	76%	25.2	7.74	10^{5}-10^{8}
8	893K	73%	27.0	7.58	10-10^{3}
9	855K	69%	24.2	7.41	10-10^{3}

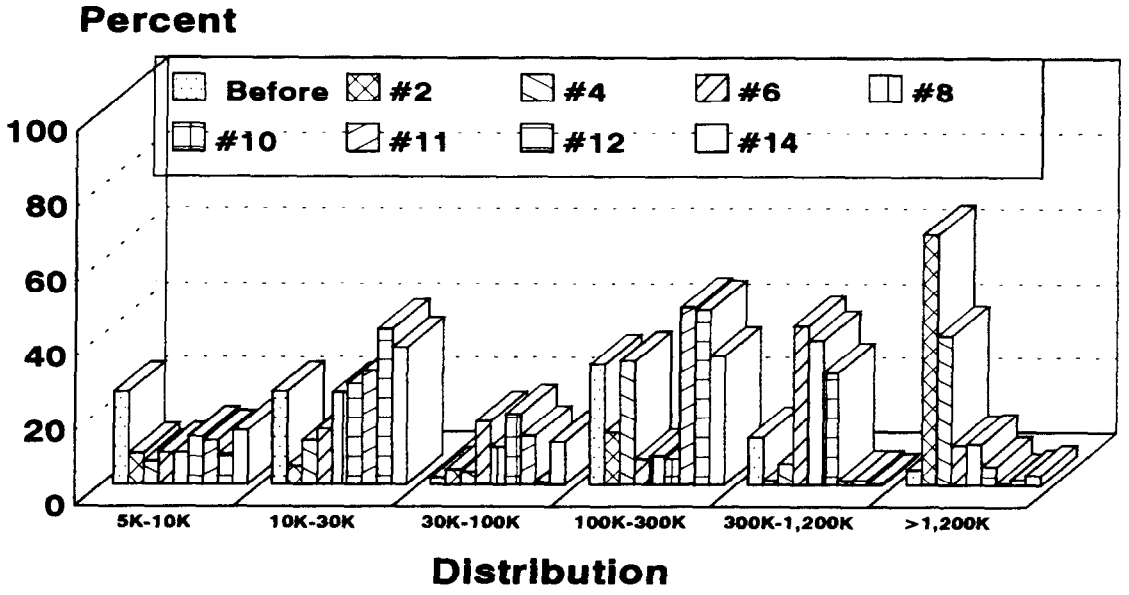

Figure 1 - Molecular Weight Distribution of Well A
Removal Treatment: Before and After

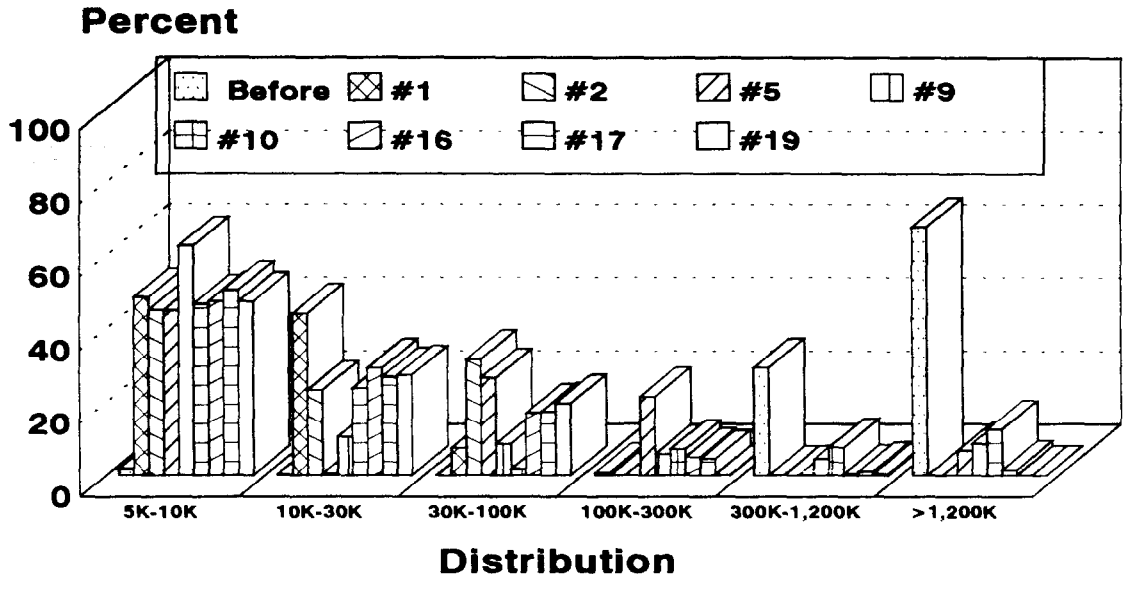

Figure 2 - Molecular Weight Distribution of Well B
Removal Treatment: Before and After

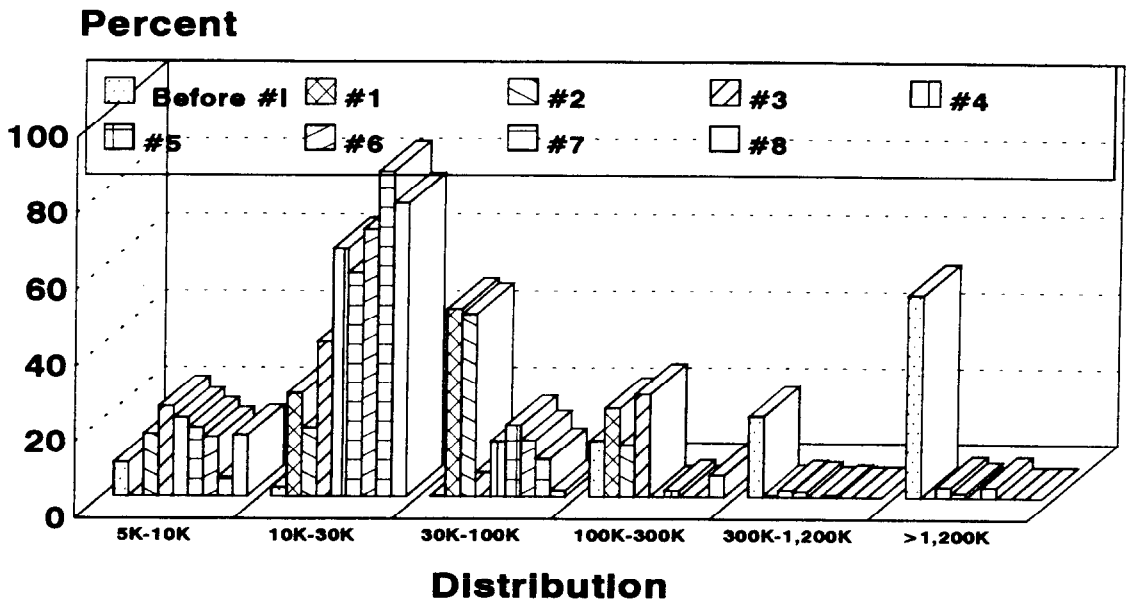

Figure 3 - Molecular Weight Distribution of Well C
Removal Treatment: Before and After

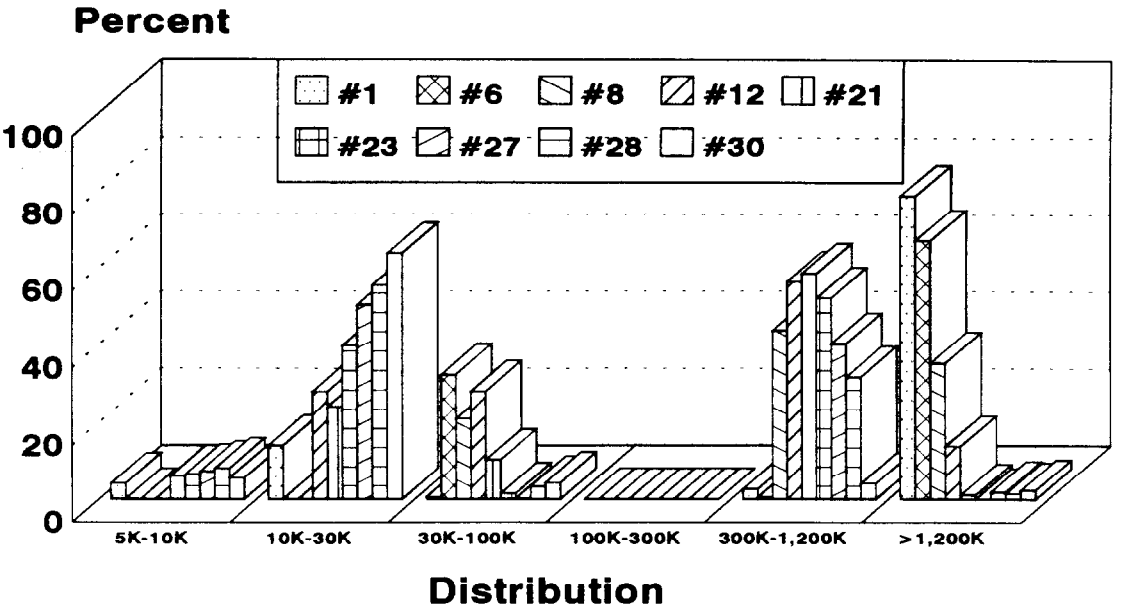

Figure 4 - Molecular Weight Distribution of Well D
40 lb/Mgal CMHPG/Zirconium @ 292°F

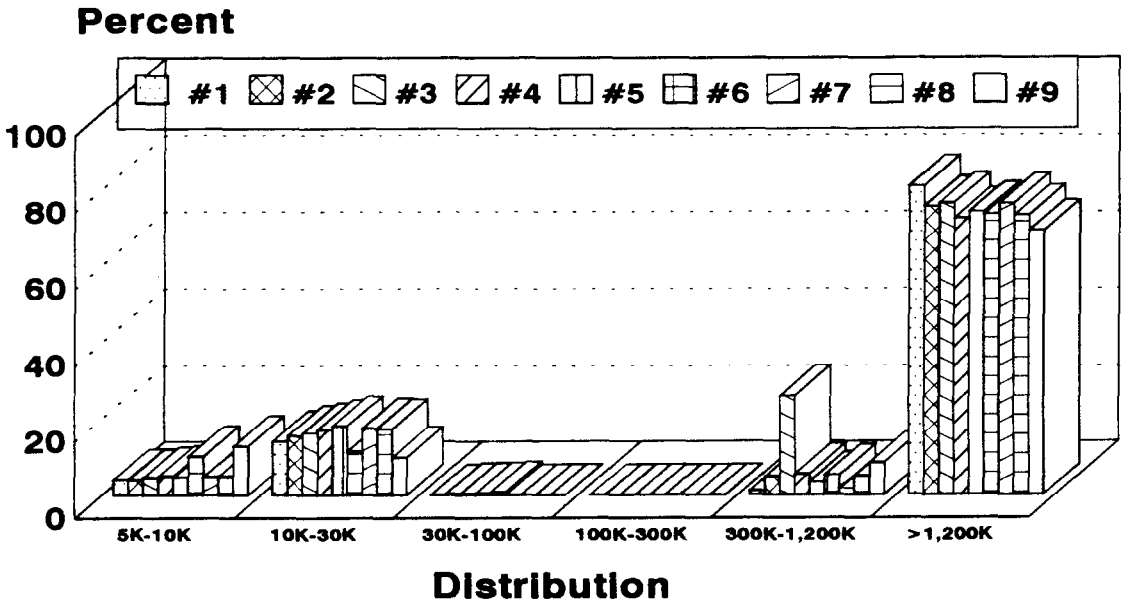

Figure 5 - Molecular Weight Distribution of Well E
30 lb/Mgal Guar/Borate @ 200°F

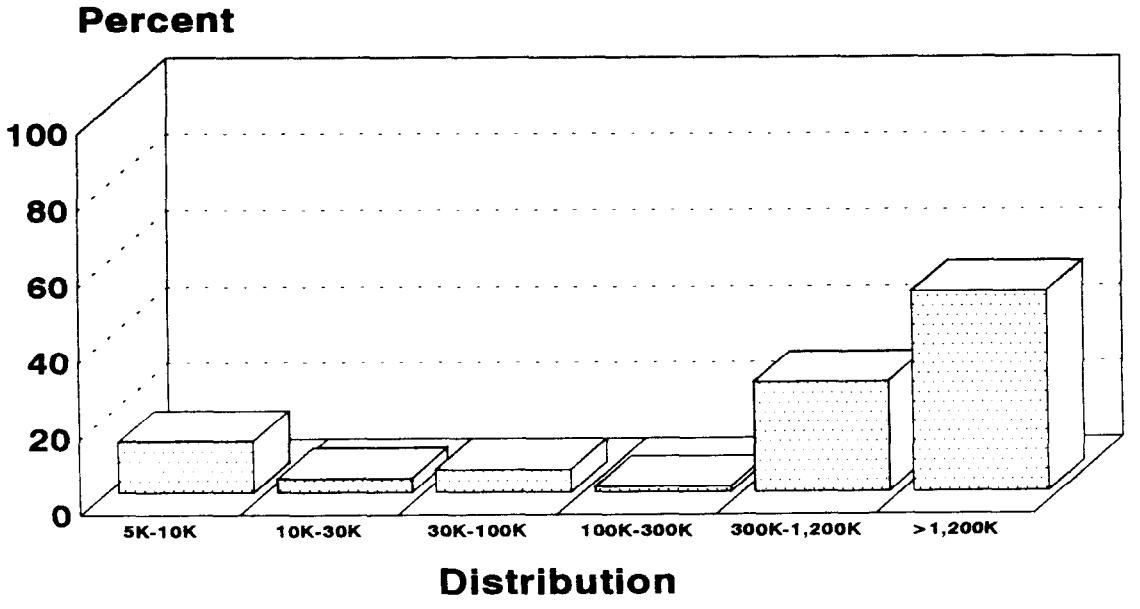

Figure 6 - Molecular Weight Distribution of Well F
Guar/Zirconium @ 265°F