

Status Report

FORMATION DAMAGE CONTROL: PARAFFIN TREATMENT TESTING AND EVALUATION

by

Feliciano M. Llave, Ph.D. and Yong Fan, Ph.D.

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

ABSTRACT

This report summarizes studies conducted to improve the control, treatment, and prevention of formation damage, particularly in the area of organic deposition. The project focuses on problems related to near-wellbore permeability changes around injection or production wells during secondary and tertiary oil production operations. The objective is to assess the available technology for controlling formation damage due to paraffin deposition and to provide the necessary research support to apply and demonstrate potential methods in field tests.

Formation damage resulting from crystallization and deposition of paraffin wax within the reservoir is a recurrent production problem. The occurrence of these problems are highly dependent upon the temperature, pressure, and flowing conditions near the wellbore and the reservoir crude compositions. Formation damage can be caused by improper well treatments such as hot oiling, non-isothermal/cold fluid injections and incompatible fluid chemistry.

This report presents the results of the laboratory screening using commercially available paraffin treatment chemicals. Experiments were conducted to evaluate the effectiveness of these solvents to remove paraffin-related formation damage. An introduction to paraffin chemistry, formation damage by paraffin, and stimulation techniques used to handle these problems is given. Results obtained from research work to measure solubility and dissolution rate of paraffin in selected solvents are presented. Efforts to initiate field site evaluations were also undertaken. A site will be selected for in-depth problem evaluation, design the field test protocol, and implementation. Supporting laboratory studies will be needed for the planned test(s).

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1.0 INTRODUCTION

1.1 BACKGROUND

Many crude oils deposit waxy materials called paraffin during production and transportation when subjected to changes in temperature and pressure. These paraffin problems cause losses of billions of dollars yearly to the petroleum industry worldwide through the cost of chemicals, reduced production, well shut-in, less use of capacity, choking of the flowlines, equipment failure, premature abandonment, and increased manpower attention (Misra et al. 1995). Figure 1-1 shows a map of the United States and the states indicated where problems (particularly related to organic deposition) have been identified. This map illustrates the geographical extent of the problem area (DOE-BPO 1995; Llave et al. 1995). Bucaram (1967) also reported that of 69 oil producing areas in 19 states, moderate to severe paraffin problems occurred in 59 of those areas. Therefore, research on paraffin-related problems is of paramount importance to oil field operations.

Paraffin is a carbonaceous material not soluble or dispersible in crude oil under the conditions where deposition occurs. These waxes normally consist of high-molecular-weight paraffin hydrocarbons including either straight-chain (normal), branched, or cyclic alkanes. They are generally very inert and resistant to attack by acids, bases, and oxidizing agents. Previous research indicated that n-paraffins are predominately responsible for the deposition problem. Compounds other than n-paraffins, especially asphaltenes and resins, occluded oil and water,

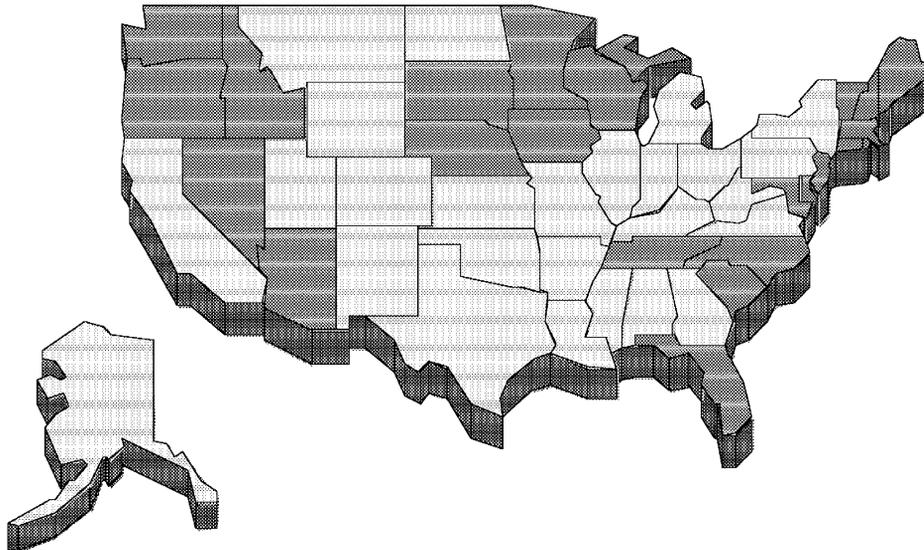


Figure 1-1 Areas Where Paraffin-Related Problems Have Been Identified

Legend: Light gray areas indicate regions with reported problems

and possibly sand and silt, have profound effects on solubility of n-paraffins (Carnahan 1989). Wax crystallization poses three problems: (1) high viscosity, which leads to pressure losses, 2) high yield stress, and (3) deposition of wax crystallization on the surface of porous media. Production and material losses will occur if the problem is not taken care of properly.

Formation damage from paraffin deposition within the reservoir can cause a significant decrease in reservoir performance. As fluids flow through the reservoir at pressure and temperatures below the cloud point of the fluid, precipitated paraffin particles are deposited within the pores of the reservoir. The cloud point is defined as the equilibrium temperature and pressure at which solid paraffin crystals begin to form in the liquid phase. Subsequently, the absolute permeability of the region of the reservoir in which deposition has occurred is reduced, which results in a decrease in reservoir flow, mostly near the wellbore (Ring et al. 1992). The three phases of paraffin deposition are: (1) solubility/precipitation of paraffin, (2) deposition of precipitated paraffin, and (3) reduction in permeability caused by paraffin deposition. A diagram illustrating paraffin precipitation and deposition in the reservoir is shown in Figure 1-2.

1.2 SCOPE OF WORK

Formation damage caused by precipitation and deposition of organic material (especially paraffin waxes) within the reservoir have gained increasing attention in oil industry. This project is focused on addressing some of these problems. This project requires BDM-Oklahoma to:

- Conduct a workshop to transfer formation damage control technology to independent producers
- Develop laboratory methods to evaluate organic-based solvents for the removal of paraffin-causing formation damages
- Design and implement field treatments by using selected chemicals and techniques developed in this research
- Provide technical support to the oil industry concerning paraffin problems

This report is organized as follows:

1. The causes of formation damages in well production operations are analyzed.
2. Stimulation methods that are currently used to handle paraffin problems are discussed.
3. The solubility evaluation of paraffin wax samples in the selected commercial solvents is presented.

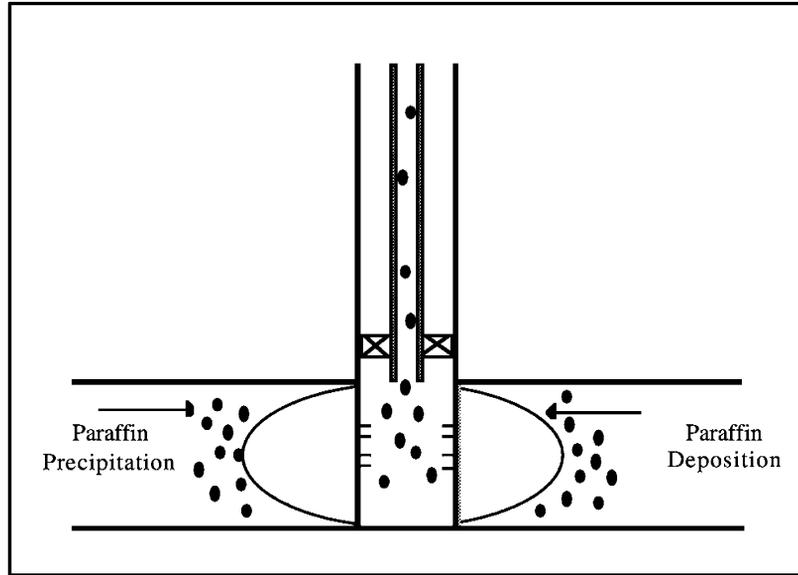


Figure 1-2 Illustration of Near-Wellbore Paraffin Precipitation and Deposition

4. The kinetics of the paraffin dissolution rate at reservoir temperatures is discussed.
5. Conclusions are drawn from this research, and future work is recommended.

2.0 DISCUSSION

2.1 CAUSES OF FORMATION DAMAGES

Almost every field operation has potential to damage well productivity. Formation damage can occur during drilling, completion, or workover. Damage can also result from routine production or injection and stimulation treatments. Fresh water invasion, solid plugging, organic material deposition, scale precipitation, fines migration, and clay swelling are common causes of low productivity. Formation damage is a very real problem that must be addressed equally by all departments within a company. It does no good for the drilling department to spend time and money doing the best job possible drilling a water-sensitive zone if the production people dump fresh water down the hole as a kill fluid (Porter 1989).

The primary cause of wax separating from crude oil is loss in solubility because of changing environmental conditions that disturb solution equilibrium (Sutton 1974; Deo 1995). Factors affecting this equilibrium include temperature and pressure changes, evaporation, and loss of dissolved gases. More specifically, the causes of formation damage are due to some field operations or unwanted flow conditions (see Table 2-1).

Injecting fluids into oil reservoirs for improving oil recovery or stimulating production is a common production practice. Most of the fluids, however, are injected at temperatures much lower than reservoir temperatures. Although these fluids are heated during injection due to the geothermal gradient, in most applications, they reach the formation at temperatures far lower than the reservoir temperatures and cool the formation in the vicinity of the wellbore. The temperature lowering frequently causes solids precipitation particularly for paraffinic crudes and for oils susceptible to asphaltene precipitation.

The use of hot oiling to clean the formation or tubing of accumulated paraffin is the most generally accepted procedure in the oil field (Barker 1989). However, hot oiling may be the most significant single cause of formation damage due to the potential plugging agents carried into the casing annulus with the oil. The paraffin or higher molecular weight alkane components of the oil are the most common cause of formation damage during hot oiling. Inorganic solids such as iron sulfide, iron oxide, clay, or sand can be damaging agents when they become oil wet during the pumping and heating process. These problems can become especially severe when these solids combine with paraffin.

Table 2-1 Causes of Formation Damage

Causes	Operations
Cold fluid injection	Acidizing job
	Fracturing job
	Water flooding
	Condensate treatment
	Fluid dump job
Cooling by gas expansion	High GOR wells
	CO ₂ floods
	NGL floods
Incompatible/contaminated fluid invasion	Hot oiling job
	Acidizing job
	CO ₂ floods
	NGL floods
	Condensate job
High flow rate through formation	Flowing well
	CO ₂ /NGL floods
	Steam floods

2.2 STIMULATION TECHNIQUES

Several methods have been proposed to remove or inhibit the damage caused by paraffin deposition. These methods include:

- Thermal
- Mechanical
- Solvent/surfactant
- Water/surfactant
- Microbial
- Combinations of the above methods

Heating the reservoir to a temperature above the cloud point is one way to restore the permeability to its initial state. This thermal method uses downhole electrical-resistance devices to heat the reservoir (Ashton 1986). Since the power must be delivered from the surface by cable, there is a large loss in power during the transport. Consequently, it limits both depth of application to about 5,000 ft and the rate of heat input to about 50 KW/hr. Stine (1984) patented a novel method for treating high viscosity oil near the wellbore as well as paraffin in the formation. In that method, called in situ hydrocracking, the formation is heated with a fire flood to 500°–900° F, hydrogen at 200–5,000 psi is forced into the formation, and the well is allowed to soak for 48 hours. The resultant hydrocracked petroleum is recovered when the well is returned to production.

The major types of chemicals available for paraffin treatment include:

- *Solvents*—Usually containing a high aromatic content, solvents are used to dissolve existing deposits. They dissolve a specific amount of paraffin depending upon the molecular weight of the paraffin, temperature, and pressure before the solvent power is exhausted.
- *Dispersants*—These compounds can break deposits up into much smaller particles which can be reabsorbed by the oil stream.
- *Paraffin detergents*—This class of surface active agents can solubilize the paraffin in oil.
- *Paraffin crystal modifiers*—Usually are selective polymers that can alter paraffin crystal growth and hence inhibit the deposition of the paraffin particles.

Use of microorganisms in the near-wellbore region of a production well can mitigate formation damage problems. Pelger (1992) reported on the results of individual treatments by using a collection of facultative anaerobic microorganisms. As reported, these neutrally charged marine organisms (in a saline solution of nutrients) are naturally occurring, nonpathogenic, nontoxic, noncarcinogenic, noncombustible, and require no permits from the Environmental Protection Agency (EPA) for storage, transportation, or disposal. Pelger claimed that by using this microorganismic technique, operating costs have been reduced by 18.1% through elimination of

chemical treatments; reduced hot oiling/watering of flowlines, wellbores, and stock tanks, and reduced mechanical cutting of paraffin from the wellbore. However, the limitations of the microbial methods are also evident, as the following requirements must be met for a microbial project (Pelger 1992):

- The survival temperature range is 34°–208°F. The microbes are dormant in the range 34°–40°F.
- The optimum temperature range is between 90°–150°F. The microbes can multiply every 20–120 minutes inside this temperature range.
- The chloride content by volume needs to be less than 15%.
- The pH needs to be greater than 3.0.
- The H₂S content in a fluid needs to be less than 1,000 ppm and 20,000 ppm in a gas.
- The formation must produce at least a trace of water, or water must be added to provide the microorganisms with an adequate means of transportation throughout the entire system in the water phase.

All in all, while several methods have been used to handle the paraffin problems, none of these methods are permanently effective. Whatever the method, the treatment should be conducted periodically depending on the reservoir conditions and production performance. Moreover, the standardization of these methods still eludes the scientific community. The effectiveness of paraffin treatment depends on the complete evaluation of the causes of the problem and the needs of the field. The critical step is that one must clearly diagnose the mechanisms and causes of the formation damages before applying these methods.

3.0 SOLUBILITY MEASUREMENT

3.1 PARAFFIN WAX AND CHEMICAL SOLVENT CANDIDATES

This evaluation of commercially-available chemical solvents attempts to focus on the wax solubility (defined as the amount of paraffin solid dissolved per 100 gram or 100 ml of solvent) and dissolution rate for a given component of paraffin materials.

Table 3–1 lists the physical properties of the selected solvents and wax samples. The paraffin wax and solvents obtained from the service and chemical companies include:

- Polywax 500 (paraffin wax used in this research) is a standard testing sample obtained from Petrolite Inc. It contains a wide range of hydrocarbons (C₁₈–C₅₄).
- Waxsol™ from Amsol Corporation is marketed as a cost-effective solvent specially designed for removal of paraffin problems in areas of wellbore, near wellbore formation, and surface/downhole production equipment and delivery and storage systems. This solvent is 100% hydrocarbons, containing high multicomponent aromatic

molecules specially refined to maximize solvation and ensure reservoir compatibility by eliminating the possibility of multiphase blockage.

- Surfatron DN-89 from Champion Technologies consists of 41% toluene, 14% petroleum naphtha, 1% isopropanol, 34% petroleum (aliphatic) distillate, and 10% other compatible components. This solvent is designed to remove paraffin deposition in all parts of a well including formation, tubular flowing lines, and production equipment.
- D-Wax[®]-10 from Baker Chemicals, a solvent for the removal of paraffin deposition in the near-wellbore and down hole, is mainly composed of xylene (<80%), mixed terpene hydrocarbons (proprietary), and methyl ethyl ketone (<5%).

The following is a list of desirable characteristics and properties of candidate treatment chemicals for application as a solvent to dissolve paraffin deposits within the reservoir:

- It should be chemically compatible with the reservoir fluids and formation materials.
- It should have sufficient solubility and moderate dissolution rate at in situ conditions.
- It should be easily removed from the reservoir once the treatment is over.
- It should be cost-effective, easy to apply, and safe to use in the field.
- It should nontoxic and not harmful to the environment.

The first characteristics listed may be the most critical. If the chemical nature of the solvent causes swelling of naturally occurring clays in the formation, thereby plugging pore channels, the treatment will be a failure. If the solvent causes migration of fines and/or clays, the success of the treatment will be nullified. If the solvent reacts with formation fluid and produces secondary precipitation, new plugging may occur.

Table 3-1 Physical Properties of Chemical Solvents and Paraffin Wax

Physical properties	Waxsol [™]	Surfatron DN-89	D-Wax [®] -10	Polywax 500
Specific gravity	0.82	0.84	0.854	0.93 @ 16°C
Vapor pressure	<0.5 psi		<5 @ 68°F	negligible
Melting point				88°C
Solubility in water	insoluble	insoluble	insoluble	insoluble
Viscosity		2-3 cP		6.6 cP @99°C
pH		5.6-7	6.4-7.4	
Flash point	66°F		<60°F	>99°C
Appearance	light amber	orange/yellow	yellow	white
Odor	hydrocarbon	aromatic	pine	little/none

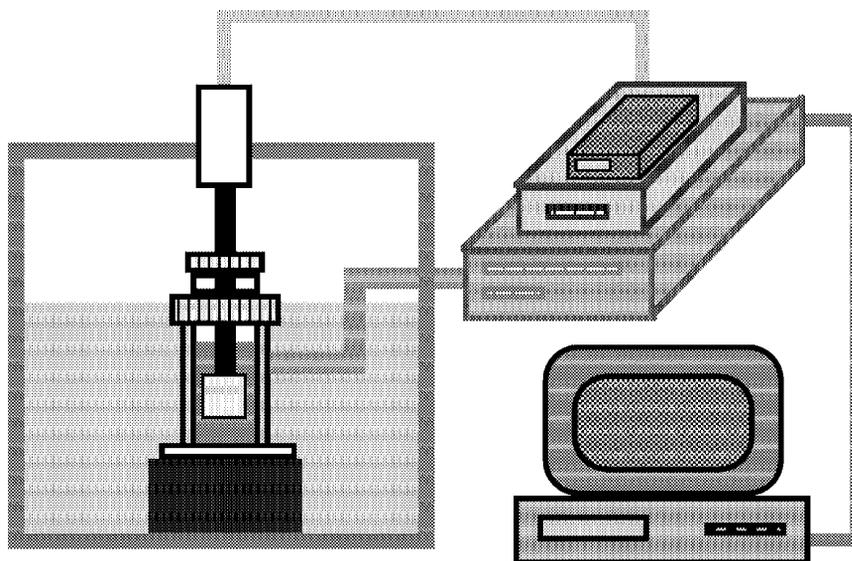


Figure 3–1 Schematic Diagram of Experimental Setup

Compatibility is therefore a critical and necessary characteristic of the chemical solvent. The ability to dissolve a sufficient amount of paraffin at a moderate dissolution rate is another important characteristic of the solvent. This capability is directly related to the efficiency when the solvent is used. Apparently, the good chemical candidate should be easy to use in the field and cost-effective. Finally, protection of the environment should be considered when chemicals are used in the field.

3.2 TEST PROCEDURE

A laboratory setup to test the solubility of paraffin wax in the selected solvent was built (see Fig. 3–1). For a typical test, the paraffin wax sample is fashioned in the shape of cylinder (OD \approx 2.75 cm, L \approx 2.65 cm) with a given surface area and is molded on the shaft of the mixing propeller. The solvent is immersed into the water bath and heated to the testing temperature. After the prescribed temperature is reached, the paraffin is immersed into a solvent (about 140 ml) at a designated stirring speed. The amount of paraffin dissolved is measured, by use of both a volumetric method and a refractive index technique, as a function of temperature, time, and rotational speed. For each of test, the samples with identical spindles were measured for every 20 minutes for the first 4 hours, then measured for every 60 minutes for the rest of the test. The test lasts for about 10 hours until the equilibrium phase is reached. In addition, a Brookfield viscometer was connected to the test setup so that the viscous behavior and cloud point of the paraffin in the solvent can be measured for the identical solution.

3.3 RESULTS AND DISCUSSION

Table 3–2 summarizes the tests conducted in this research. These experiments include evaluation of three selected solvents obtained from the different companies at different temperature levels. Some of the tests are still ongoing and are not presented in this report.

Figures 3–2 to 3–7 show typical dissolution curves (expressed as the amount of the paraffin wax dissolved as a function of time) at a given temperature in selected solvents. The dissolution rate varies with time. In the initial time steps, the dissolution rate is much faster and very evident because the paraffin wax is exposed to the "fresher" solvent than in the later time period. As the experiment progresses, the solution approaches its saturated state. Beyond this point, the equilibrium between the dissolution and precipitation is reached. The maximum amount of wax dissolved at equilibrium is defined as the solubility limit at the given experimental conditions.

Figures 3–2 to 3–4 show temperature dependence of the capacity of each of the solvents tested to dissolve the target paraffin wax sample. The highest amounts of wax dissolved in these solvents occurred at the 60°C level. An orderly decrease in dissolved amount is observed as temperature decreases.

Table 3–2 Summary of Tests Conducted on Selected Solvents

Solvents	Test at 35°C	Test at 50°C	Test at 60°C
Waxsol TM	X	X	
Surfatron DN-89	in progress	X	X
D-Wax [®] -10	X	X	X

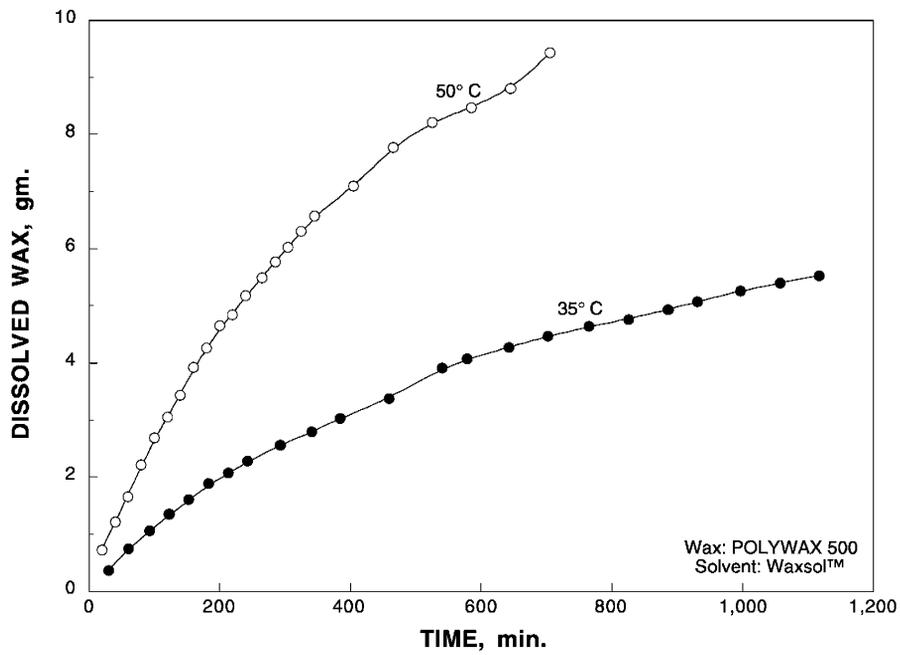


Figure 3-2 Amount of Paraffin Wax Dissolved in Waxsol™

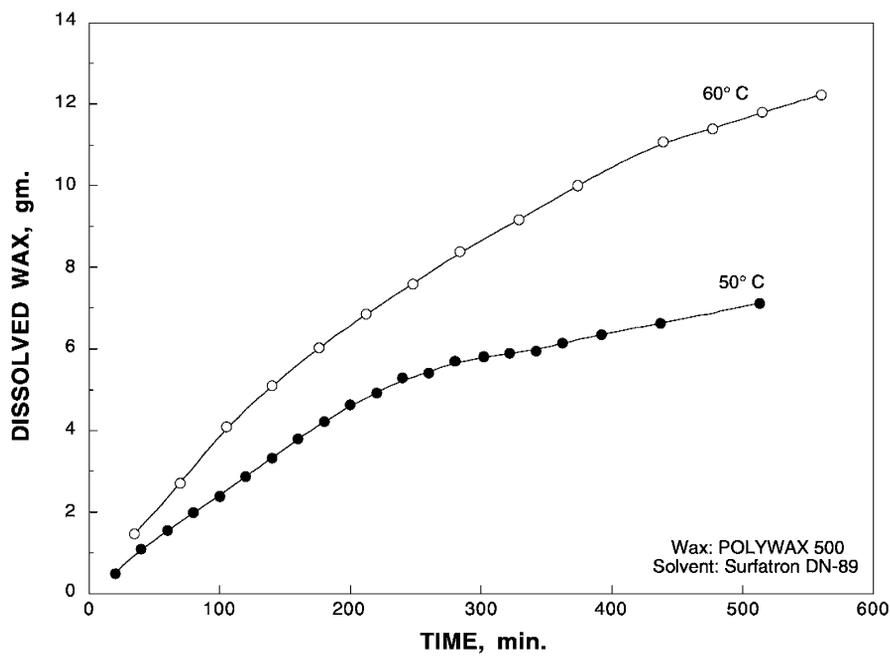


Figure 3-3 Amount of Paraffin Wax Dissolved in Surfatron DN-89

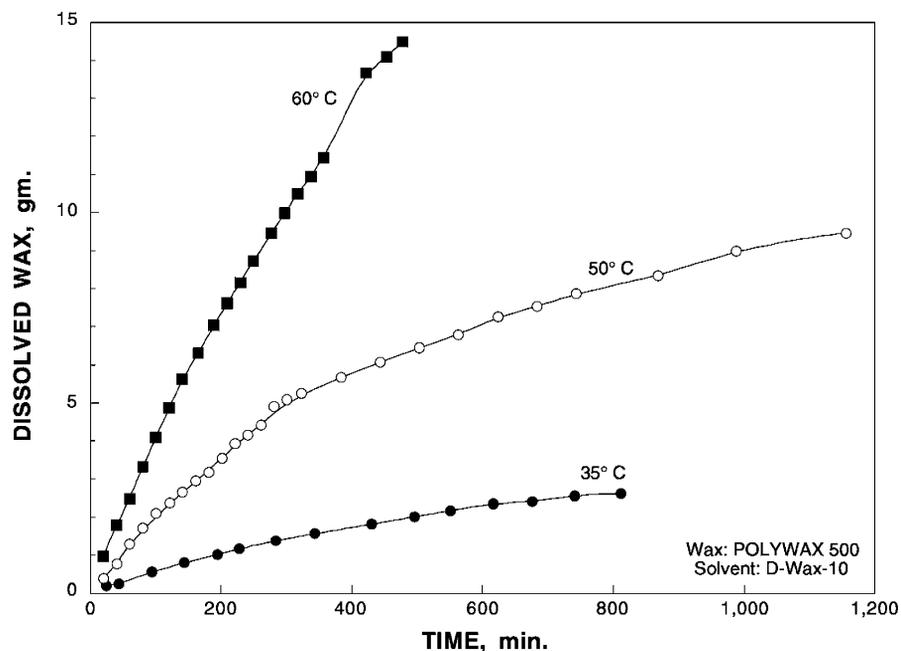


Figure 3-4 Amount of Paraffin Wax Dissolved in D-Wax[®]-10

Figures 3-5 to 3-7 show a comparison of the amount of wax dissolved in each of the solvents with respect to temperature. Figure 3-5 shows a comparison of the Waxsol[™] and D-Wax[®]-10 at 35°C. The results indicate that Waxsol[™] was more effective in dissolving comparable amounts of wax than the D-Wax[®]-10 under the conditions tested. Figure 3-6 shows the results of the experiments conducted at 50°C. This figure compares the three solvents: D-Wax[®]-10, Surfatron DN-89, and Waxsol[™]. A similar comparison was made at 60°C. Figure 3-7 shows the results of the experiments using Waxsol[™] and Surfatron DN-89 at this temperature. The results indicate that the Waxsol[™] was more effective in dissolving wax than the Surfatron DN-89 at the elevated temperatures. The overall results indicate that the samples tested yielded the following order of increasing capacity to dissolve paraffin wax (under comparable conditions):

$$\text{D-Wax}^{\text{®}} 10 < \text{Surfatron DN-89} < \text{Waxsol}^{\text{™}}$$

Additional experiments will be conducted to provide a direct comparison at 35°C and at 60°C for all three solvents. Only the experiments at 50°C were completed and analyzed for the three solvents tested. Repeat experiments will also be conducted for comparison and verification. Additional treatment chemicals from other manufacturers will also be requested and tested for comparison.

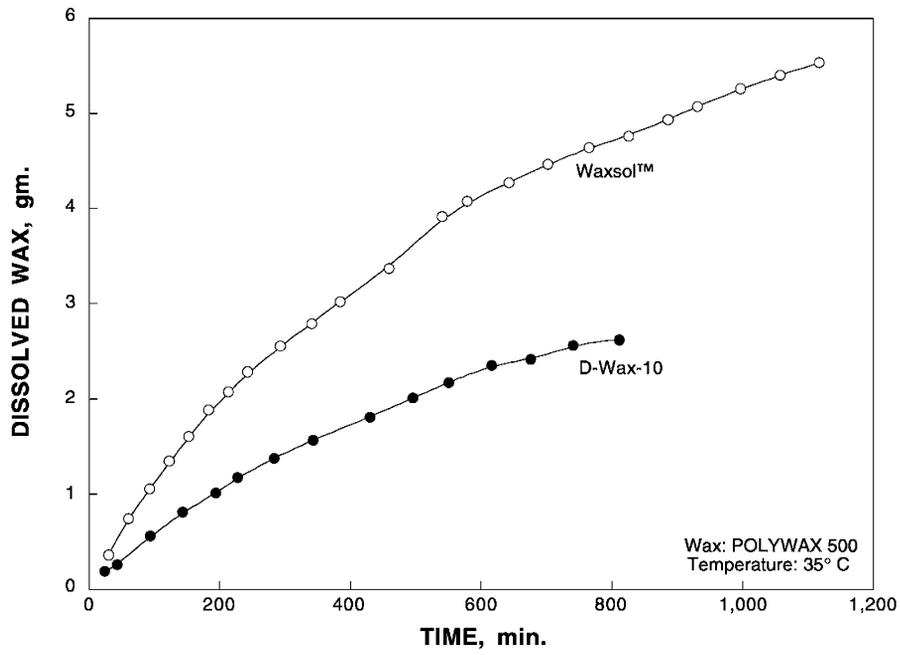


Figure 3-5 Amount of Paraffin Wax Dissolved in the Solvents at 35°C

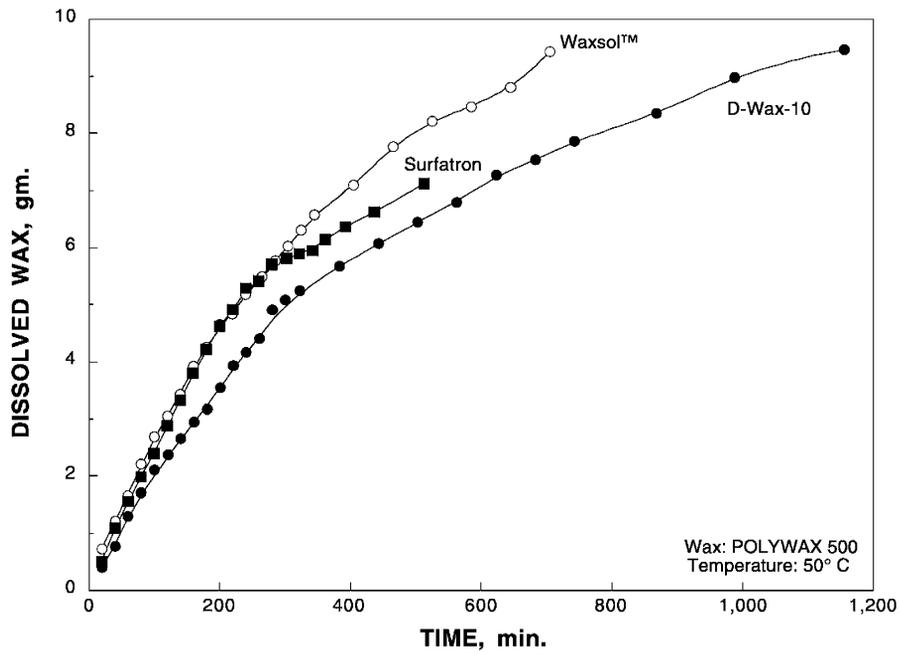


Figure 3-6 Amount of Paraffin Wax Dissolved in the Solvents at 50°C

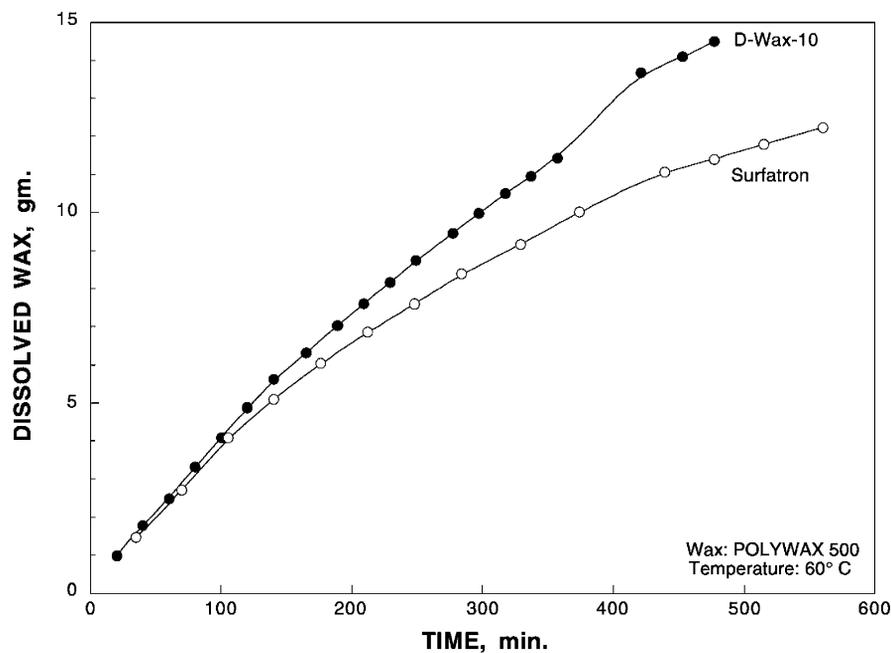


Figure 3-7 Amount of Paraffin Wax Dissolved in the Solvents at 60°C

3.4 KINETIC APPROACH

The kinetic approach to paraffin dissolution attempts to determine the rate at which paraffin wax is dissolved in each of the selected solvents. This information is important in designing and supervising a field treatment, and also indispensable for laboratory evaluation of solvent candidates. The dissolution of paraffin solids in a chemical solvent is a physical process because no new materials are produced. The process consists of three consecutive, but separate steps including:

1. Solvent mass from the bulk of the solution "diffuses" to the surface of the solid due to the convection and concentration gradient.
2. Wax surface dissolution process occurs.
3. Dissolved particles "leave" the solid surface, mixing with the bulk solution.

Initial efforts evaluated the kinetics of dissolution rate of Polywax 500 in selected solvents. The observed dissolution rate of paraffin in the solvent (a physical process) was postulated as a *pseudo-chemical reaction*; therefore, the principle of chemical reaction can be applied. The proposed general rate expression is as following:

$$-\frac{d[C_o - C]}{dt} = k [C_o - C]^n \quad (3-1)$$

where C_o and C are solubility limit and concentration of the paraffin in the solvent, respectively; k is dissolution rate constant (a function of temperature only); and n is the dissolution reaction order.

This rate expression has taken the physical process of dissolution into account, i.e., the dissolution rate approaches zero as the paraffin concentration in the solution reaches the solubility limit. The integrated forms of the dissolution rate are as follows:

$$-\frac{1}{1-n} \left[\frac{1}{(C_o - C)^{n-1}} - \frac{1}{(C_o)^n} \right] = kt \quad \text{for } n \neq 1 \quad (3-2)$$

and

$$-\ln \left[\frac{C_o - C}{C_o} \right] = kt \quad \text{for } n = 1 \quad (3-3)$$

Equation (3-3) indicates that a plot of the natural logarithm of the normalized concentration gradient with time is a straight line. The dissolution rate constant can be obtained from the slope of the line. Figure 3-8 shows a sample plot of the dissolution rate as a function of time, cast in the form of equation (3-3).

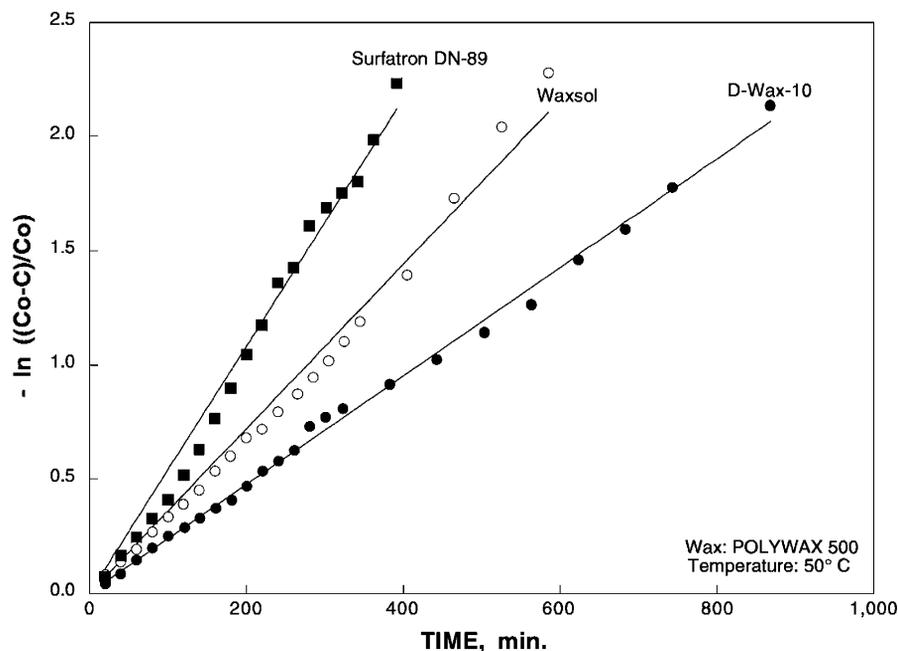


Figure 3-8 Rate of Dissolution of Paraffin in Solvents at 50°C

The test data at 50°C indicated that the dissolution of the paraffin in the solvents appears to follow a pseudo-first order reaction form. The results of the experiments at the other temperature levels will be analyzed. The additional data will be correlated with respect to the pseudo-first order reaction proposed to evaluate if the "fit" is acceptable.

3.5 CONCLUSIONS

Based on the results obtained in this research, the following conclusions are drawn:

1. The experimental setup has been built to measure the solubility and dissolution rate of paraffin wax in the selected chemical solvents at different temperatures. The results suggest the use of chemical solvents has the promising potential benefit to clean the paraffin deposition in near-wellbore formations.
2. The solubility of paraffin in the solvents is a strong function of temperature and increases considerably with an increase in temperature. Of the three solvents evaluated, Waxsol™ appears to have the greatest solvation capacity for Polywax 500 at a temperature of 50°C. The following order of increasing amount of paraffin wax dissolved was observed:

$$\text{D-Wax}^{\text{®}} 10 < \text{Surfatron DN-89} < \text{Waxsol}^{\text{™}}$$

3. Dissolution of paraffin solid in the solvents is a physical process consisting of three consecutive steps. This process is postulated to be treated as a pseudo-chemical reaction and thus the chemical principle is applicable in the paraffin dissolution. An initial analysis of the results indicates that the dissolution rate can be expressed as a first order reaction.
4. Paraffin precipitation and deposition are governed by a very complex set of factors including temperature, pressure, flowing conditions, and crude compositions. In most cases, formation damage is caused by improper well treatments such as hot oiling, non-isothermal/cold fluid injections, and incompatible fluid chemistry. These problems can amount to significant production and material losses if not taken care of properly.
5. A number of stimulation methods have been proposed to control and remediate formation damage from paraffin deposition. However, none of these methods are permanently effective. Whatever the method, the treatment should be conducted periodically depending on the reservoir conditions and production performance. Moreover, the standardization of these methods still eludes the scientific community.

4.0 PRELIMINARY FIELD SITE EVALUATION

One focus of this project is to identify candidate field test sites for in-depth evaluation of problems related to formation damage and control attributable to paraffin deposition. Preliminary screening centered on two possible candidates which exhibited some problem during production operations.

The Naval Petroleum Reserve No. 3 (NPR-3), commonly referred to as the Teapot Dome Field north of Casper, Wyoming, is a typical Rocky Mountain reservoir that can be characterized as a heterogeneous, highly fractured and faulted sandstone formation. Based on initial discussions with the operator in Natrona County, Wyoming, the 2nd Wall Creek and Shannon formations appear to be the formations that exhibit paraffin problems.

The characteristics of the paraffinic crude oil in these reservoirs are somewhat similar. The 2nd Wall Creek reservoir contains a 36° API gravity crude oil of 0.16% sulfur, whereas the Shannon reservoir contains a 35.2° API gravity crude, with 0.14% sulfur. Table 4-1 lists some of the characteristics of the two reservoirs.

Different methods to treat paraffin-related problems have been applied in this field. More recent effort by the operator centers on the application of downhole magnets and microbial-based systems. Additional operational information will be needed to evaluate the extent of their problem. Preproposal meetings with the operator have been arranged for early September.

Table 4–1 Reservoir Data for NPR-3

Reservoir Data	2nd Wall Creek	Shannon
Lithology	Sandstone	Sandstone
Porosity, %	16	16
Permeability, md	North: 10; South: 50	50 avg.
Avg. Pay Thickness	25 feet	30 feet
Drive Mechanism	Solution gas	Gravity drainage
Salinity, ppm	11,000	6,450
Oil, ° API	36°	35.2°

The second possible site is also in the Rocky Mountain area. The site has been under an active waterflood for the last six years. Crudes from this site have been analyzed to have a relatively high paraffin content. Preliminary discussions with the operator provided insight on concern over both production and injection operations. Results of an analysis using wellbore temperature profile calculations showed the strong possibility of paraffin/wax deposition occurring and the potential for reduced injectivity (formation damage) in the vicinity of the wellbore during operations. Current efforts to control the problem include regular treatment of production wells using a microbial system. Plans are being developed to facilitate work on evaluating their current operations pertaining to paraffin deposition control.

5.0 FUTURE WORK

Plans are:

- To develop a thermodynamic model to predict the paraffin precipitation and deposition at in situ conditions, and complete the evaluation of solubility and dissolution rate for the chemical solvents.
- To build a dynamic core flooding system to examine the solvent flow in the formation rock at controlled temperature and pressure conditions.
- To develop linear and radial mathematical models to simulate the solvent flow in the porous medium.
- To implement field tests and treatment evaluation.

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