TUBING CLEANOUT STUDIES WITH AN ENVIRONMENTALLY SAFE PICKLING FLUID

Sandra L. Berry, Joel L. Boles and Scott G. Nelson
BJ Services Company

ABSTRACT

A slightly acidic (pH of 5.8-6.3) Tubing Cleanout Fluid has been evaluated in the laboratory and in the field for use as a pickling fluid to replace traditional acids. This fluid's capability to remove iron scales on coiled tubing, prevent or delay rust from reforming, clean up commercial pipe dope, and reduce corrosion rates, was compared to traditional hydrochloric acid in laboratory studies. This fluid performed as efficiently as 15wt% hydrochloric acid at 140°F in removing rust/iron scale, as well as pipe dope from the workstring with lower corrosion rates. Furthermore, a work string treated with this fluid retained a protective coating that resisted re-oxidization for up to 30 days. Also, several case histories detailing the product's field use for pickling completion workstrings are discussed.

INTRODUCTION

Benefits of Pickling Treatments

Stimulation and completion treatments, whether it be an acidizing job, gravel pack job, or completion with a solids free completion brine, are conducted to remove formation damage and/or increase the productivity index of the near wellbore formation region. Industry standards recommend a preventative preliminary pickling treatment to assure the best possible success from the stimulation or the completion treatments. Pickling treatments are utilized to remove iron scale, rust, pipe dope and other hydrocarbon deposits from the downhole tubulars and surface equipment such as tanks, pumps, and flowlines which prevents introduction of these contaminants into the production zone thereby affecting future well productivity. Benefits of conducting a pickle treatment to remove these tubing contaminants are iron control, sludge prevention, removal of damaging pipe dope, elimination of spent acid entering the formation, improvement in stimulation results, and reduction in overall treatment cost. Introduction of these contaminants into the formation can result in loss of well productivity if they enter the targeted oil-producing zone. Therefore, efforts to remove these contaminants before any chemical treatments begin are very advantageous.

Iron control is probably the most important aspect of a pickling treatment.[1,2] The dissolution of iron scale or rust solubilized during a HCl acid treatment will form both ferric and ferrous ions in acid. Formation damage can occur from any acid treatment when ferric ions precipitate as gelatinous ferric hydroxide after the pH of spent acid increases above 1-2. [2,4]

Another concern regarding iron contamination of the main acid stimulation treatment is the formation of damaging asphaltic sludges created by the incompatibility of spent acid and certain formation crude oils. This asphaltic sludging is induced by both ferric and ferrous iron contamination. However, ferric iron has been shown to induce a more severe sludging tendency than the ferrous ion. Therefore, an important key in prevention of asphaltene sludging tendencies between the spent acid and the formation crude oil is to minimize the ferric and ferrous iron content of the acid. [2,4]

Another common contaminant present in downhole tubulars and casing is excess pipe dope. Most pipe dopes are composed of very high viscosity grease with friction reducers, plating materials, and heavy metals such as lead, zinc, and copper. Thread compounds are used between the drill pipe sections during preparation of the drill string to seal tubular joints and reduce thread wear. Unfortunately, pipe dope also does an excellent job of plugging and sealing gravel packs, formations, and perforations if allowed to enter into the producing zone. Pipe dope compounds as well as iron scales and rust may seal the formation, making acid treatment extremely difficult and inefficient.[3,4]

Historically, cleaning these pipe dope materials and other hydrocarbon deposits had been conducted with a stable dispersion of an organic solvent such as a terpene-based solvent, xylene or toluene in an HCl acid system. The stable dispersion of the solvent allows aromatic solvent to dissolve the pipe dope or organic deposits and enables the internal acid phase of the dispersion to react with the rust and iron scale compounds. [2,3,4]

Traditional Pickle Acid Treatments

Traditional pickle acid systems typically consist of a 10 to 15% hydrochloric acid. Additives such as corrosion inhibitors and surfactants to facilitate a water-wet surface during the pickling treatment are also included. In addition, a solvent is typically added to aid in the removal of pipe dope, asphaltenes, waxes, and hydrocarbon deposits. Pickling treatments are pumped down the inside of the tubulars and are reversed circulated back to the surface without fluids being lost to the formation. This procedure removes the most soluble and the formation-damaging contaminants from the wellbore and ensures these materials are not placed in the formation as a possible cause of reduced well productivity

Environmental Concerns of Traditional Pickling Systems

Typical pickling acid treatments, in Gulf of Mexico deepwater applications, cost approximately $10,000 for the purchase, transportation, rigtime, neutralization process, and disposal cost. Upon return to the surface after treatment, pickle acids are classified as a hazardous material with a pH range of less than 0 and require neutralization at the rigsite with soda ash before disposal.[1,2,3] These returns pose safety, environmental, and liability risks associated with handling and disposing of a hazardous material. Use of an Environmentally Safe Pickling fluid to remove rust, iron scales, pipe dope and hydrocarbon deposits would be very advantageous. Furthermore, pickling returns from an Environmentally Safe Pickling fluid would eliminate soda ash neutralization procedures at the rigsite, as well as safety, environmental, and liability risks associated with handling hazardous acid returns.

This paper discusses the laboratory evaluation of an Environmentally Safe Pickling fluid to remove rust, iron scale, and pipe dope, to delay or prevent the reoxidation on treated metal surfaces, for corrosion rates at typical wellbore temperatures, and the fluid's environmental /safety issues. It will also discuss two case histories of field application of the Environmental Friendly Pickling fluid system for removal of typical tubing contaminants.

BACKGROUND

Tubing Clean-Out Fluid Composition

Based on the safety, environmental, and liability concerns associated with acid pickling, a Environmentally Friendly Pickling fluid system was developed based on iron phosphate plating technology. This solution has a slightly acidic pH, both as the initial product and the pickle return system. It is an environmentally favorable formulation capable of deoxidizing a wide variety of metals through its chelating action. It contains phosphonate source in a acidic form, buffering agents and a catalyst to speed up the reaction at low temperatures.[3,5]

The Environmentally Friendly Pickling agent reacts with iron scale, rust, and the metal surface itself producing a protective film that coats the metal and helps to prevent or delay the rate of reoxidation through a passitivity mechanism. Depending on the treatment variables, a gunmetal silvery or dark gray surface is formed which will last for several days or weeks.[3,5] Results of the laboratory studies conducted on the Environmentally Friendly Pickling fluid system are discussed in the following text.

EXPERIMENTAL

Derusting Properties

Laboratory studies to evaluate the derusting properties of the Environmentally Safe Pickling fluid were conducted with heavily oxidized coiled tubing coupons. HCl pickle acid (15wt%), 5vol% and 12vol% Environmentally Safe Pickling fluid systems were evaluated for iron scale removal efficiency. Initial dimensions and weights were taken on each of the coupons before incubation of the metal samples in its respective pickling fluid formulation for a one-hour period at 140°F with constant stirring. After the one-hour incubation at 140 °F, the treated coupons were removed from the test solution, washed with water, and allowed to dry. After drying, the final weights were measured and the difference in the initial/final weights was determined to calculate the percent iron scale removal. Results of the derusting studies on the coiled tubing coupons show that the 12vol% Environmentally Safe Pickling fluid was as efficient in removing rust from the surface of the oxidized coiled tubing coupon as the 15wt% HCl pickle acid solution. The 12vol% Environmentally Safe Pickling fluid removed 2.24% rust and the 15wt% HCl acid removed 2.43% rust from the coiled tubing surface.

Furthermore, another indicative tool that can be used in determining the derusting efficiencies of a pickling fluid system is the fluid's total and soluble iron concentrations, as well as Total Suspended Solids (T.S.S.) content after treatment. Analyzes of the final pickling fluid systems were conducted for initial/final pH values and total /soluble iron content by DCP (Direct Coupled Plasma) spectroscopy. Total Suspended Solids content was also conducted according to API Recommended Practices for Analysis of Oilfield Water and calculated in milligrams per liter. Results of the analysis on the final pickling fluid systems show the 12vol% Environmentally Safe Pickling fluid contains 3,088 mg/l of total iron in comparison to 2,796 mg/l of iron in the 15wt% HCl acid system. Furthermore, the pH values of the Environmentally Safe Pickling fluid after pickling were measured in the 6.3 to 6.5 range. Table 1 of this paper details the calculated percent rust removal of the coupons, as well as the final pickling fluid's initial/final pH value, iron contents, and T.S.S. concentrations. Figures 1–4 of this report show the coiled tubing coupons and pickling fluid solutions before and after a pickling treatment.

Rust Prevention Properties

After laboratory pickling, the four treated coiled tubing coupons were incubated at atmospheric conditions of 90°F with relative humidities of 90% for a period of 21 days to evaluate the rate of reoxidation. Observations of the treated coupons after the 21-day incubation period show the coiled tubing coupons treated with the Environmentally Friendly Pickling fluid exhibit a gunmetal silver or light gray surface, which delays the rate of reoxidation or rust formation in comparison to the metal surfaces treated with 15wt% HCl. Figure 5 of this report show a photo of the treated metal coupons after the 21-day incubation period at atmospheric conditions.

Pipe Dope Removal Studies

Tubing cleanout procedures in which pipe dope or organic deposit are likely to be present require the addition of a solvent to facilitate the removal of this material. The Environmentally Friendly Pickling fluid with and without the addition of a terpene-based solvent was evaluated in comparison to 15 wt% HCl pickle acid with and without terpene solvent in removing three commercially available pipe dope materials. L80 coupons were coated with each respective pipe dope and reacted with the test solution for a one-hour period at 140°F with constant stirring. After the one-hour incubation period, the coupon was removed from the test solution and allowed to air dry overnight.

After drying, the coupons were weighed and their physical characteristics were recorded. The initial and final weights of the coupons were utilized to determine the pipe dope removal efficiency. Surface observations were recorded and photos were taken. These studies will indicate pipe dope removal and underlying rust removal efficiency.

Results of the pipe dope removal studies with the three pipe dope materials are represented in Tables 2, 3, and 4 of this paper and show that pipe dope cleaning removal was the most efficient with the Environmentally Friendly Pickling fluid containing 25vol% terpene-based solvent. Figure 6 shows an initial pipe dope coupon, as well as coupons after treatment, with 15wt% HCl and 12vol% Environmentally Friendly Pickling fluid containing 25vol% terpene-based solvent. Figure 7 shows a graphical representation of the pipe dope removal efficiencies of the four test fluid formulations with the three pipe dope materials. Pipe dope removal efficiency tests using 15wt% HCl acid with and without the addition of 25vol% terpene solvent yielded less efficient pipe dope removal results with oil-wet metal surfaces. Furthermore, use of the Environmentally Friendly Pickling fluid with the addition of a terpene-based solvent resulted in complete removal of the three commercial pipe dope materials and left the metal surface water-wet.

Corrosion Studies

Corrosion studies on the Environmentally Friendly Pickling fluid and 15wt% HCl acid were conducted at typical wellbore temperatures with and without corrosion inhibitors for five hours using N80 and coiled tubing metals. Weight losses of the test coupons during the test period were determined and the corrosion rates in lb.ft2/test period were calculated.

Under these test conditions, the maximum recommended allowable corrosion rate for N80 metal is 0.05 lb.ft2/test period with a Pitting Index no greater than 1. The maximum recommended allowable corrosion rate for the coiled tubing samples is 0.02 lb.ft2/test period with a Pitting Index no greater than 0. The corrosion rate determinations for the Environmentally Friendly Pickling fluid and the 15wt% HCl acid systems were compared to determine the relative corrositivity of the Tubing Clean-Out Fluid.

The corrosion rate studies conducted with the Environmentally Friendly Pickling fluid at 140°F for five hours with N80 and coiled tubing metals show both systems exhibit very low corrosion rates without the addition of corrosion inhibitor at 140°F. Table 2 details the results of the corrosion studies in lb.ft2/test period and the Pitting Index of the final metal samples. The calculated corrosion rate for the Environmentally Friendly Pickling fluid at 140 °F for 5 hours with coiled tubing metal was 0.028 lb.ft2 per test period and 0.0245 l.ft2 per test period for N80 carbon steel.

Table 3 of this paper details the corrosion rate studies of the 15wt% HCl and the 12vol% Environmentally Friendly Pickling fluid at 180°F for five hours with N80 and coiled tubing metals. As the incubation temperature increased, the corrosion rates of the 15wt% HCl acid system with no corrosion inhibitor for a five-hour test period increased to 0.2364 lbs.ft2/test period for the N80 metal and 0.0791 lbs.ft2 for the coiled tubing metal. Corrosion rate test data for the 12vol% Environmentally

Friendly Pickling fluid with no corrosion inhibitor at 180°F show rates of 0.0555 lb.ft2/test period for N-80 metals and 0.073 lb.ft2/test period for coiled tubing samples. At 180°F, the 12vol% Environmentally Friendly Pickling fluid without corrosion inhibitor shows a N80 metal corrosion rate one-fifth that of 15wt% HCl containing no corrosion inhibitor.

Acceptable corrosion inhibition for the 12vol% Environmentally Friendly Pickling fluid was achieved for the desired metals (N80 and coiled tubing samples) at 180°F for the test period of five hours. Results of the five-hour corrosion studies at 180°F conducted with the 12vol% Environmentally Friendly Pickling fluid inhibited with 1 gpt of CI #4 show corrosion rates of 0.0098 lb.ft2/test period for the N80 metals and 0.0168 lb.ft2/test period for the coiled tubing samples.

CASE HISTORIES

Introduction

To date, several hundred pickling treatments employing the Environmentally Friendly Pickling fluid as the pickling agent have been performed in the United States and South America. Furthermore, in the last six months, over 15 pickling job applications with the Environmentally Friendly Pickling fluid have been conducted in land wells in the Permian Basin. Bottomhole static temperatures of the wells pickled with the Environmentally Friendly Pickling fluid have ranged from 128°F to 150°F and the true vertical depths have ranged from 7,200 ft to 8,480 ft. A wide range of tubular metals (J55, L-80 and N-80) with 2 7/8-in. outer diameter and 6.5#/ft were subjected to treatment with the Environmentally Safe Pickling fluid.

Two case histories with the Environmentally Safe Pickling fluid are presented in this paper. Case history #1 discusses the pickling of the tubulars in an oil well in Argentina and shows the results of the treatment with the initial and final pickling fluid changes and total iron content of the final pickling solution.

Case history #2 of this report details an innovative application of the Environmentally Safe Pickling fluid in pickling a subsea fuel gas line system located in the Campos Basin Offshore Brazil area. This study will show photos of the fluid changes as well as the total iron content of the samples collected during pickling treatment.

Case History 1

This South America oil well is located in the Chihuido de la Sierra Negra Field of Neuquen, Argentina. It was reported to have a packer set at 931 meters with 2 7/8-in., 6.5 lbs/ft tubing. Before the main acid stimulation treatment was pumped, the tubulars were pickled with a 10vol% Environmentally Safe Pickling fluid with the addition of a 3vol% pine-based solvent system for removal of rust, iron scale, pipe dope and hydrocarbon deposits. The 10vol% Environmentally Safe Pickling fluid in water with the addition of a 3vol% pine-based solvent system was pumped and displaced to the packer at a rate of 0.15-0.25 BPM. The well shut-in time and total soaking time for the Environmentally Safe Pickling fluid was 2.5 hours. After the 2.5-hr soaking time, the Environmentally Safe Pickling fluid was returned to the surface for pickle returns fluids observations and total iron content. The total iron content of the returned Environmentally Safe Pickling fluid was determined to be 1,140 mg/l of iron. Figure 7 of this report show photos of the Environmentally Safe Pickling Fluid from the rig return flowlines at the initial pickle return stage and when approximately 2/3 of the total pickling fluid volume had been displaced to the surface. Figure 8 of this report show photos of the Environmentally Friendly Safe Pickling fluid initially before pumping, as well as middle and final stage return samples. These photos show the dramatic change in the Environmentally Safe Pickling fluid from the initial colorless and clear samples to the very rusty and oily final pickling fluid returns.

Case History 2

This pickling application of the Environmentally Safe Pickling fluid took place in the Campos Basin Offshore of Brazil and involved rust and iron scale removal inside a subsea fuel gas system line to the turbine generators A/B, turbine generators C/D, and to the water collector/skim vessel. The objective of this treatment was to remove iron scale and rust deposits created in the fuel gas system lines after the previous water-pressure test. Heavy rust and iron scales had formed in the lines after water had been inadvertently left in the line for many months without being drained or dried.

The pickling treatment was conducted with a 15vol% Environmentally Safe Pickling fluid based on the BHST of 80°F in the subsea fuel gas line system. The fluid was pumped in the subsea lines and allowed to soak for a 12-hr reaction time. However, due to the heavy rust conditions of the lines and the number of interconnected lines needing treatment, several pickling treatments were conducted to assure complete derusting of the subsea fuel lines. During the pickling treatment, samples of the pickle returns were obtained and the iron content levels were monitored. Figure 9 of this paper shows a photo of the pickle fluid returns and the iron content analysis of those fluid samples during the treatment. Review of the sample's characteristics and the resulting iron contents of the pickle returns shows a dramatic increase in the iron content up to 7,000 ppm during the pickling procedure. When the return pickling fluid had a iron concentration below 5 ppm, this iron concentration was considered to be an acceptable level for industrial cleaning and the treatment was considered successful. After the treatment, the subsea fuel lines were flushed with fresh water and dried with nitrogen to help prevent any further oxidization.

REFERENCES

1. Pourciau, R. "Case History: Internally Coated Completion Workstring Successes", paper SPE 77687 presented at SPE Annual Technical Conference and Exhibition, San Antonio, Tx., 29 September-2 October 2002.

2. Smith, B.: "Proper Treatment of Tubulars Key to Iron Control," Southwestern Petroleum Short Course, 1990.

3. Curtis, James and Kalfayan, Leonard, "Improving Wellbore and Formation Cleaning Efficiencies With Environmental Solvents and Pickling Solutions," paper SPE 81138 presented Latin American And Caribbean Petroleum Engineering Conference in Port-of-Spain, Trinidad, West Indies, 27-30 April 2003.

4. Nasr-El-Din, H.A. et al: "Lessons Learned From Acid Pickle Treatments of Deep/Sour Gas Wells," paper SPE 73706 presented at the SPE International Symposium and Exhibition on Formation Damage Control, Lafayette, Louisiana, 20-21 February 2002.

5. Berry, S.L., Boles, J.L., and Di Lullo, G.F.: "Evaluation of a Safe, Slightly Acidic Tubing Clean-Out Fluid," paper SPE 84125 presented at the SPE Annual Technical Conference and Exhibition in Denver, Colorado, 5-8 October 2003.

ACKNOWLEDGMENTS

The authors wish to thank BJ Services for permission to publish this paper. The authors would also like to acknowledge the contributions of the BJ Services Brazil and Argentina Laboratory and pumping services personnel for their assistance in providing field data.

Table 1
Summary of Environmentally Friendly Pickling Fluid Derusting Performance

Pickling Fluid	%Rust Removal	T.S.S. (mg/l)	Initial/Final pH	Total Iron Unfiltered/Filtered (mg/l)
1	2.43	91	-0.44/-0.45	2,796 / 2,359
2	1.55	69	6.06/6.91	1,995 / 1,832
3	2.24	97	6.02/6.46	3,088 / 3,017
4	2.07	104	6.03/6.27	2,652 / 2,450
5	2.07	83	6.03/6.31	2,897/2,657

1. 15 wt% HCl acid + 2 gpt nonionic surfactant + 1 gpt corrosion inhibitor
2. 5 vol% environmentally friendly pickling system in fresh water
3. 12 vol% environmentally friendly pickling system in fresh water
4. 12 vol% environmentally friendly pickling system in fresh water + 1 gpt corrosion inhibitor #2
5. 12vol% environmentally friendly pickling system in fresh water + 1 gpt corrosion inhibitor #1

Table 2 Pipe Dope #1 Removal Tests					
Test Solution	Weight			% Removal	Comments
	Initial	After Pipe Dope	After Test Reaction		
15% HCl + 2 gpt Surfactant + CI #1	37.203	38.3162	37.2216	98.30%	Pipe dope and rust still present
12 vol%. Tubing Clean-out Fluid in water + CI #1	38.966	40.8905	39.0324	96.5%	Oil-wet surface with pipe dope and rust
12 vol%. Tubing Clean-out Fluid + 25vol% Solvent + CI #1	37.561	37.8175	37.419	>100%	No pipe dope or rust with water-wet surface
15% HCl + 2 gpt Surfactant + CI #1+25 vol% Solvent	38.056	39.5305	37.9002	>100%	Oil-wet surface with pipe dope present

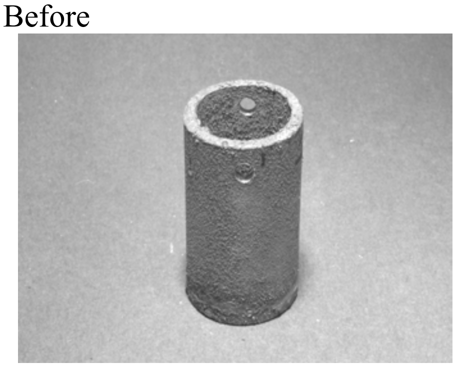
Table 3 Pipe Dope #2 Removal Tests					
Test Solution	Weight			% Removal	Comments
	Initial	After Pipe Dope	After Test Reaction		
15% HCl + 2 gpt Surfactant + CI #1	38.880	39.102	38.8494	>100%	Oil-wet surface with pipe dope present
12 vol%. Tubing Clean-out Fluid in water + CI #1	37.138	37.3225	37.134	>100%	Pipe dope and rust present on surface
12 vol%. Tubing Clean-out Fluid + 25vol% Solvent + CI #1	37.889	38.2221	37.8843	>100%	No pipe dope or rust with water-wet surface
15% HCl + 2 gpt Surfactant + CI #1+25 vol% Solvent	37.366	37.5554	37.3899	87.6%	No pipe dope but rust remains on the surface

Table 4 Pipe Dope #3 Removal Tests					
Test Solution	Weight			% Removal	Comments
	Initial	After Pipe Dope	After Test Reaction		
15% HCl + 2 gpt Surfactant + CI #1	38.828	39.5836	38.831	99.7%	Rust and pipe dope still present
12 vol%. Tubing Clean-out Fluid in water + CI #1	37.014	37.7987	37.4601	43.1%	Pipe dope present with an oil-wet surface
12 vol.% Tubing Clean-out Fluid with 25 vol%. Solvent with CI #1	37.876	38.5915	37.8622	>100%	No pipe dope or rust with water-wet surface
15% HCl + 2 gpt Surfactant + CI #1+25 vol% Solvent	37.373	37.9043	37.4373	88.0%	Rusty coupon with small amount of pipe dope present

Table 5 Five Hour Corrosion Tests @ 140 °F					
Test conditions - 140 °F under atmospheric conditions					
Metal #1 - N-80					
Metal #2 – Coiled tubing					
Time = 5 hours					
Fluid #1 –15wt% HCl + 2 gpt surfactant					
Fluid #2 – 5 vol% Tubing Clean-out Fluid in water					
Fluid#3 – 12 vol% Tubing Clean-out Fluid in water					
Fluid#4 – 5 vol% Tubing Clean-out Fluid + 20% Biocide #1 in water					
Fluid System	Metal #	Inhibitor	Quantity	Corrosion Rate ($lb.ft^2$ / test period)	Pitting
1	1	None	None	0.0306	1
1	1	CI #1	1 gpt	0.0033	Tr-1
1	2	None	None	0.0135	1
1	2	CI #1	1 gpt	0.0039	1
2	1	None	None	0.0145	Tr-1
2	2	None	None	0.0142	1
3	1	None	None	0.0245	Tr-1
3	2	None	None	0.028	Tr-1
4	1	None	None	0.0366	0-Tr
4	2	None	None	0.0437	1

Table 6 Five Hour Corrosion Test @ 180 °F					
Test conditions - 180 °F under atmospheric conditions					
Metal #1 - N-80					
Metal #2 – Coiled tubing					
Time - 5 hours					
Fluid #1 – 15 wt% HCl + 2 gpt surfactant					
Fluid #2 – 5 vol% Tubing Clean-out Fluid in water					
Fluid #3 - 12% Tubing Clean-out Fluid in water					
Fluid System	Metal #	Inhibitor	Quantity	Corrosion Rate Lbs/ft2/test period)	Pitting
1	1	None	None	0.2364	3-Edges
1	1	CI #1	1 gpt	0.0089	Tr
1	2	None	None	0.0791	1\2
1	2	CI #1	1 gpt	0.0063	1
2	1	None	None	0.025	Tr-1
2	2	None	None	0.0327	0
3	1	None	None	0.0555	Tr
3	2	None	None	0.073	0
3	1	CI #4	1 gpt	0.0098	0-Tr
3	2	CI #4	1 gpt	0.0168	0

Before



After

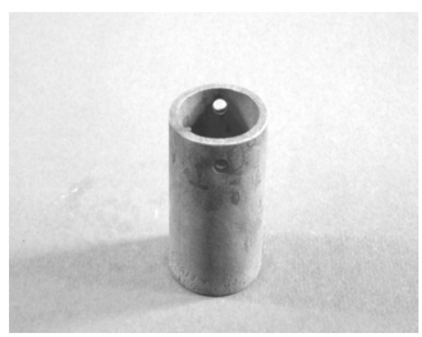


Figure 1 - Coiled Tubing Coupon Pickled with 15wt% HCl acid + 2 gpt Surfactant + 1 gpt CI

Before After

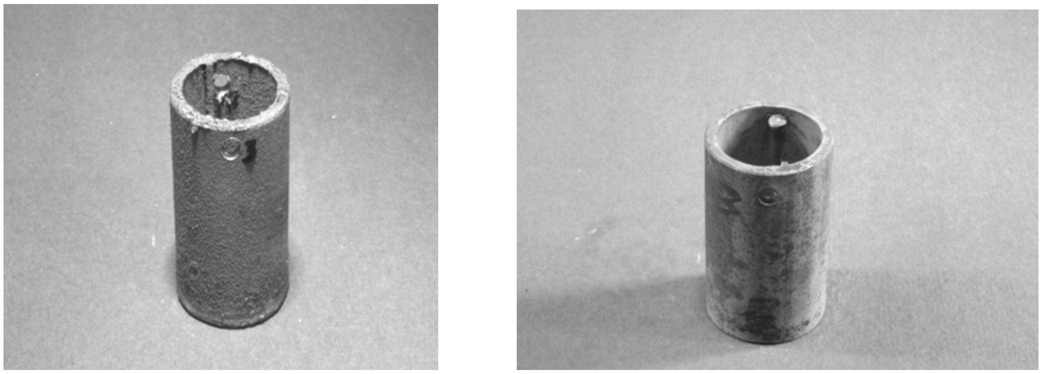


Figure 2 - Coiled Tubing Coupon Pickled with 12vol% Environmentally Friendly Pickling Fluid In Fresh Water

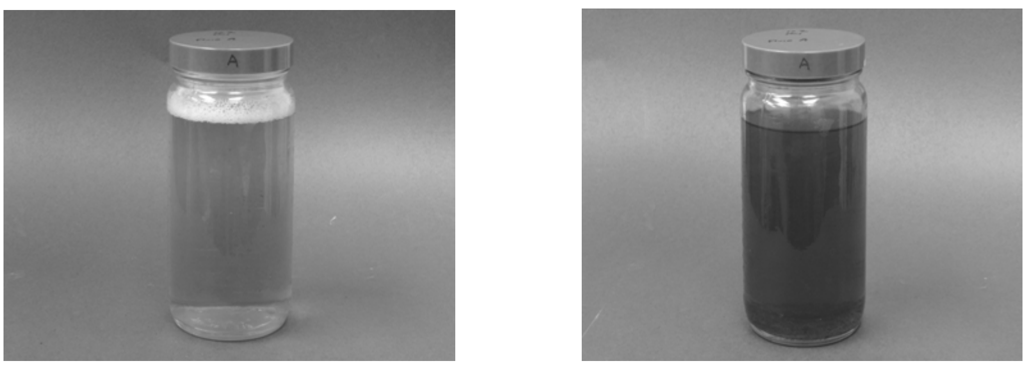


Figure 3 - 15wt% HCl Acid Before and After Pickling

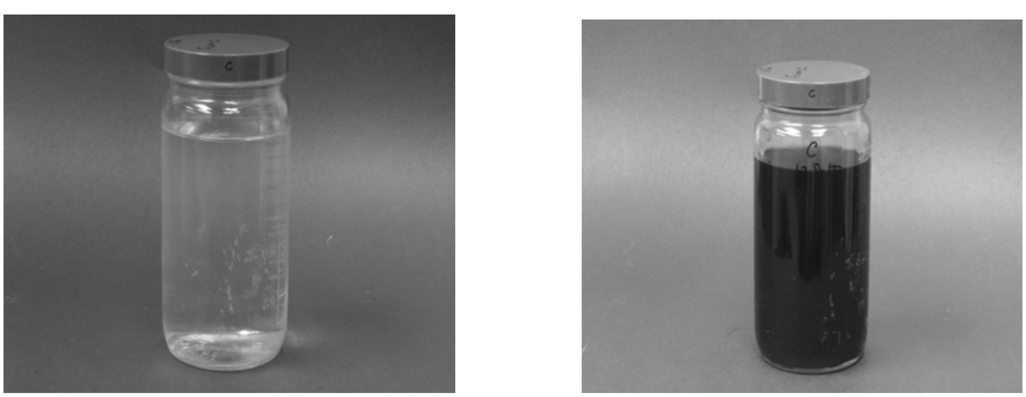


Figure 4 - 12vol% Environmentally Friendly Pickling Fluid Before and After Treatment

Figure 5 - Treated Coupons After 21 Day Incubation At Atmospheric Conditions

L80 Coupons Coated With Pipe Dope Before Testing

L80 Coupon Treated With 15% HCl Acid + 25vol% Terpene Based Solvent

L80 Coupon Treated With 12vol% Environmentally Friendly Pickling Fluid + 25vol% Terpene Based Solvent

Figure 6 - Pipe Dope Removal Studies With HCL Acid and Environmentally Friendly Pickling Fluid With Terpene Solvent

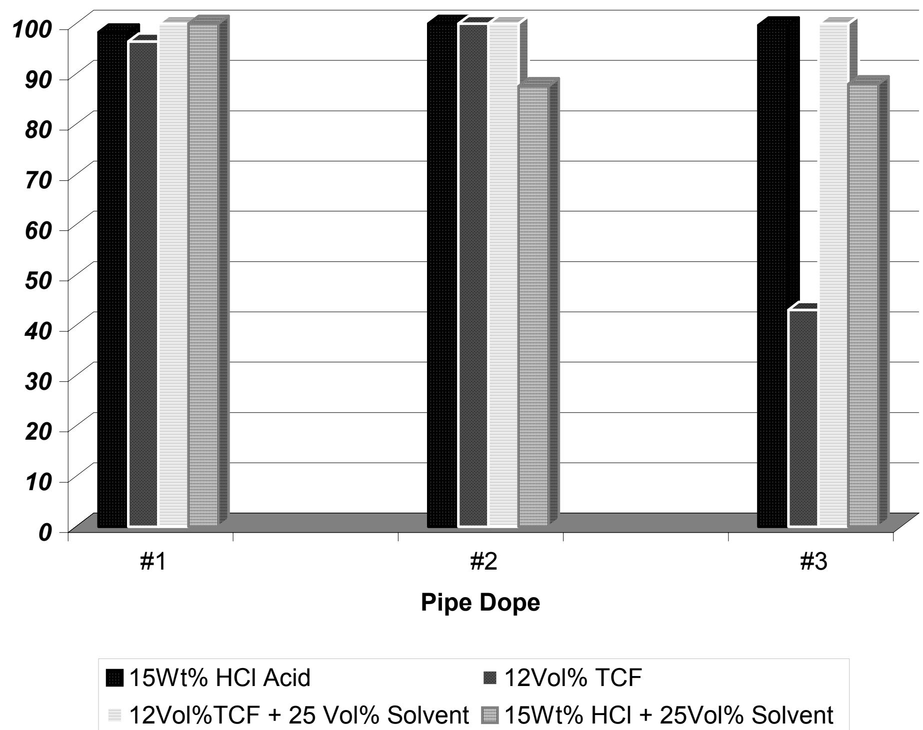
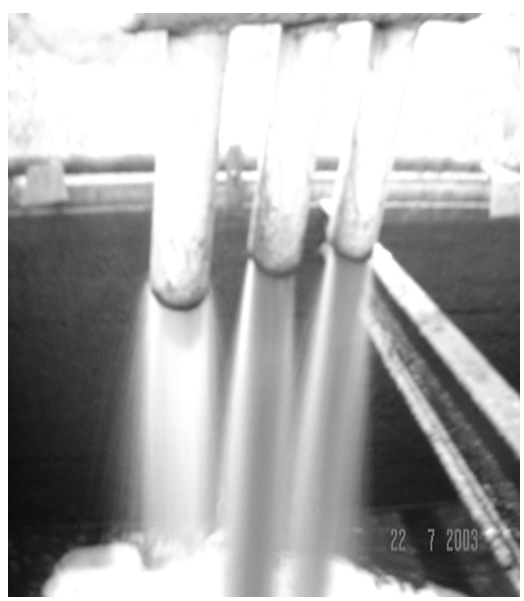


Figure 7 - Comparative Pipe Dope Removal Performance

Initial Pickling Returns



Middle Fluid Volume Returns

Figure 8: Return of Tubing Clean-Out Pickling Fluid From Rig Flowlines

Before treatment **Middle reverse circulation** **Final stage reverse circulation**

Figure 9 - 10% Tubing Clean-Out Fluid with 3%vol Pine Based Solvent

Clean Up Samples

Sample 0: Fresh Water (450 ppm Fe)
Sample 1: First Pickling (7000 ppm Fe)
Sample 2: Second Pickling (5000 ppm Fe)
Sample 3: Third Pickling (2250 ppm Fe)
Sample 4: Fourth Pickling (600 ppm Fe)
Sample 5: Fifth Pickling (2 ppm Fe)

Figure 10 - Pickle Return Samples From Subsea Fuel Gas Line System