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Novel Polymeric Phosporate Scale Inhibitors for improved Squeeze

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CONTENTS

NOVEL POLYMERIC PHOSPHONATE SCALE INHIBITORS FOR IMPROVED SQUEEZE TREATMENT LIFETIMES

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SUMMARY

New patented chemistry has provided an exciting discovery which may be used to reduce costs in scale squeeze applications. Phosphonomethylated polyamines (PMPA's) have been found to possess outstanding adsorption-desorption properties which generate long squeeze lifetimes. This paper describes the core-flood tests and modelling work, which highlight these properties, plus additional scale inhibiting performance studies to demonstrate the all-round capabilities of this chemistry for squeeze treatments.

An example of a PMPA is used to show the extremely viable adsorption and desorption isotherms. These illustrate the efficient way in which the desorption occurs to minimise the chemical in the returns with a benefit of reduced chemical content in the discharge. The PMPA also demonstrates that both polymer and phosphonate properties can be embraced in a single product (e.g. dual scale control mechanisms) confirming that this chemistry is, indeed, a true polymeric phosphonate.

INTRODUCTION

The problems associated with mineral scale deposition in oil-field production systems are well known throughout the oil industry and these have been researched in depth by many people. Likewise the application of scale inhibitors in order to mitigate the problems caused by mineral scale deposition is also well established. The use of squeeze treatments to provide a means of applying scale inhibitors to production wells in order to prevent scale in the perforation tunnels, production string, valves, chokes, etc. is also well established in the oil industry.[1,2]

Indeed the use of scale inhibitor squeeze treatments in North Sea oil production has been practised for more than fifteen years, with varying degrees of success, depending upon the local conditions.[3,4]

Therefore we do not propose to discuss these issues in any detail. There are many literature references to squeeze treatment mechanisms and related topics. [5, 6, 7, 8, 9, 10]

Over the years since the introduction of the squeeze treatment technique, a range of products based upon differing types of chemistry evolved. Originally Aminotrimethylene Phosphonic Acid (ATMP) was commonly used especially in the USA and Middle East. Diethylene Triamine Phosphonic Acid (DETAP) based products have become widely used in North Sea applications, as well as Poly Phosphino Carboxylic Acid (PPCA) based products. In addition Polyvinyl Sulphonic Acid (PVSA) based products have been introduced for some severe barium sulphate problems.

The aim of this paper is to provide information and outline the benefits available from a novel chemistry invented, developed and patented by Petrolite Corporation. Such products are based upon the result of phosphonomethylation of polyamines to give PMPA's (Phosphono - Methylated - Poly - Amines). These products combine the attributes of both polymers and phosphonates for the inhibition of mineral scales in oilfield brine systems.

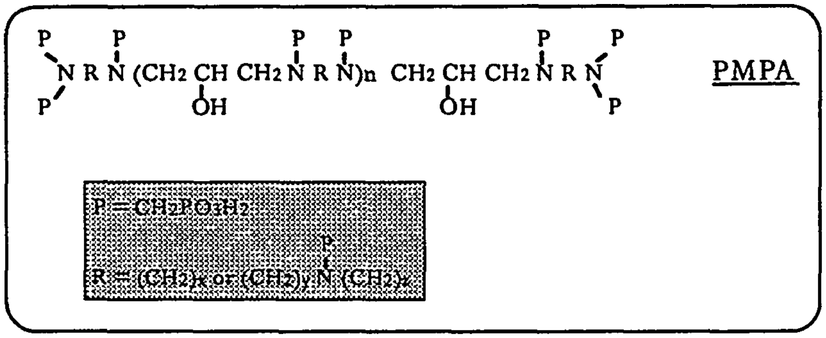


Fig. 1

These PMPA type products were originally investigated and developed by Petrolite Corporation in the mid 1980s for the treatment of sulphate problems at conditions of low pH (usually 4.0 to 5.0). For example, barium sulphate in North Sea and USA oil production as well as calcium sulphate in USA fields where CO_2 flooding was utilised, (i.e. under conditions of low pH of the brine matrix in certain reservoirs where high levels of dissolved carbon dioxide in the brine caused depressed pH conditions).

As part of the continued research into this type of work programme the excellent adsorption/ desorption characteristics of these products were also noted [11]. This, of course, offered the potential for longer squeeze treatment lifetimes and follow-up work was carried out by Petrolite Ltd in Liverpool to more closely evaluate the potential of PMPA for squeeze treatment applications. This included core-flood work and modelling exercises, in-house and in collaboration with the Oilfield Scale Research Group (OSRG) of the Department of Petroleum Engineering at Heriot-Watt University, Edinburgh, U.K.

This paper describes the major findings of that work and shows the potential benefits to be obtained by the squeeze treatment application of PMPA.

The outcome is a commercial scale inhibitor with superior squeeze properties which has the substantial potential for further development in the future, tailoring the chemical to address specific application requirements.

EXPERIMENTAL

The development and identification of a suitable PMPA for long squeeze life was highlighted from various test protocols ranging from core-flooding to performance tests and compatibility studies. These are dealt with in turn in this section.

Core-flood and Squeeze Modelling Studies

A series of core-flood experiments were carried out using basic core-flood equipment and utilising techniques established by the OSRG, Department of Petroleum Engineering at Heriot-Watt University. Core-flood studies and modelling (Squeeze IV) was also carried out by OSRG for Petrolite Ltd. as part of the study.

The experimental test is based on the Heriot-Watt regime for coreflood testing as outlined in their Experimental Procedure Manual, section 3.3. This experiment is designed to check that the chemicals can be squeezed in sandstone formations without any plugging effects and also to give an indication of their potential squeeze life in comparison to each other.

The three inhibitors used in the test are identified as follows:-

PMPA - A phosphonated polyamine scale inhibitor.

DETAP - A penta phosphonated amine scale inhibitor.

PPCA - A polyphosphinocarboxylic acid scale inhibitor.

The brine used was a synthetic North Sea brine with composition given in Table 1.

The core material used in the test was a resin coated Clashach sandstone core supplied by Heriot-Watt University. The three products were tested simultaneously in three separate cores at 70°C.

Table 1

Ion	mg/L	Salt	g/l Salt
Bicarbonate	75	$NaHCO_3$	0.1
Sulphate	2,800	Na_2SO_4	4.14
Chloride	20,500		
Potassium	400	KCl	0.76
Calcium	400	$CaCl_2.2H_2O$	1.47
Magnesium	1,300	$MgCl_2.6H_20$	10.86
Strontium	8		
Sodium	11,447	NaCl	25.72
TDS	36,855		

Core-flood Brine Composition

Core-flood Test Protocol
The core-flood test rig was set-up as outlined in Fig. 2.

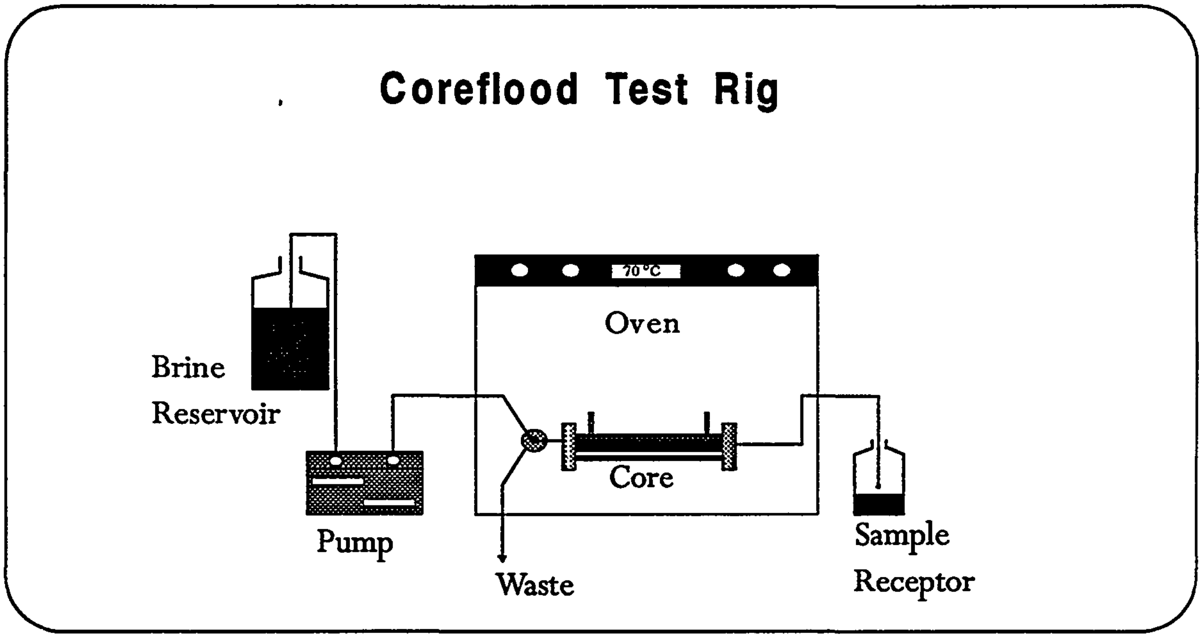


Fig. 2

The 3 cores were initially conditioned with synthetic sea water at room temperature. A lithium tracer study was then performed to determine the accurate pore volume (PV) of the cores. This was achieved by passing a 5 ppm lithium trace in synthetic sea water through the cores, collecting the effluents, and measuring the lithium concentration. The shape of the lithium return curve also indicates if there is a problem with the internal condition of the cores.

The permeability of the cores was then determined by measuring the differential pressure across the cores at various brine flow rates, constructing a $\Delta p/Q$ plot (where Δp = Differential Pressure and Q = flow rate) and measuring the slope of the line. The permeability of the core can then be calculated.

The cores were then saturated with inhibitor solutions at room temperature. For this test, all inhibitor strengths were standardised at 5 % active inhibitor concentration in synthetic sea water. The saturation of the cores was stopped after ca. 7 pore volumes when full inhibitor absorbance had been achieved and the influent and effluent level of inhibitor was equal.

The cores were then "shut in" at 70°C for 16 hours.

After the "shut in" the inhibitor desorption was performed by flushing the cores with synthetic sea water, collecting the effluents via a fraction collector and measuring the inhibitor concentration. The inhibitor concentration was plotted against pore volume to show the comparative desorption return profiles of the three scale inhibitors.

The inhibitor concentrations were all determined by plasma emission spectroscopy.

The complete comparative desorption return profiles of the three products is shown in Fig. 4.

The following aspects were investigated as a comparative study between the existing (DETAP) and the new chemistry (PMPA):-

1. External Heriot-Watt University (OSRG) core-flood studies using Clashach core samples to construct isotherms followed by Squeeze IV modelling.

2. In-house (Petrolite) core-flood on Clashach core samples followed by modelling studies using the Heriot-Watt University (OSRG) test methodology and the Squeeze IV computer modelling capability.

3. Further modelling using Squeeze IV comparing Clashach and field cores adsorption/desorption data derived by Heriot-Watt University, OSRG. The field core data was derived under reservoir condition type testing per the Heriot-Watt OSRG manual.

Core-flood/Modelling - Results & Conclusions

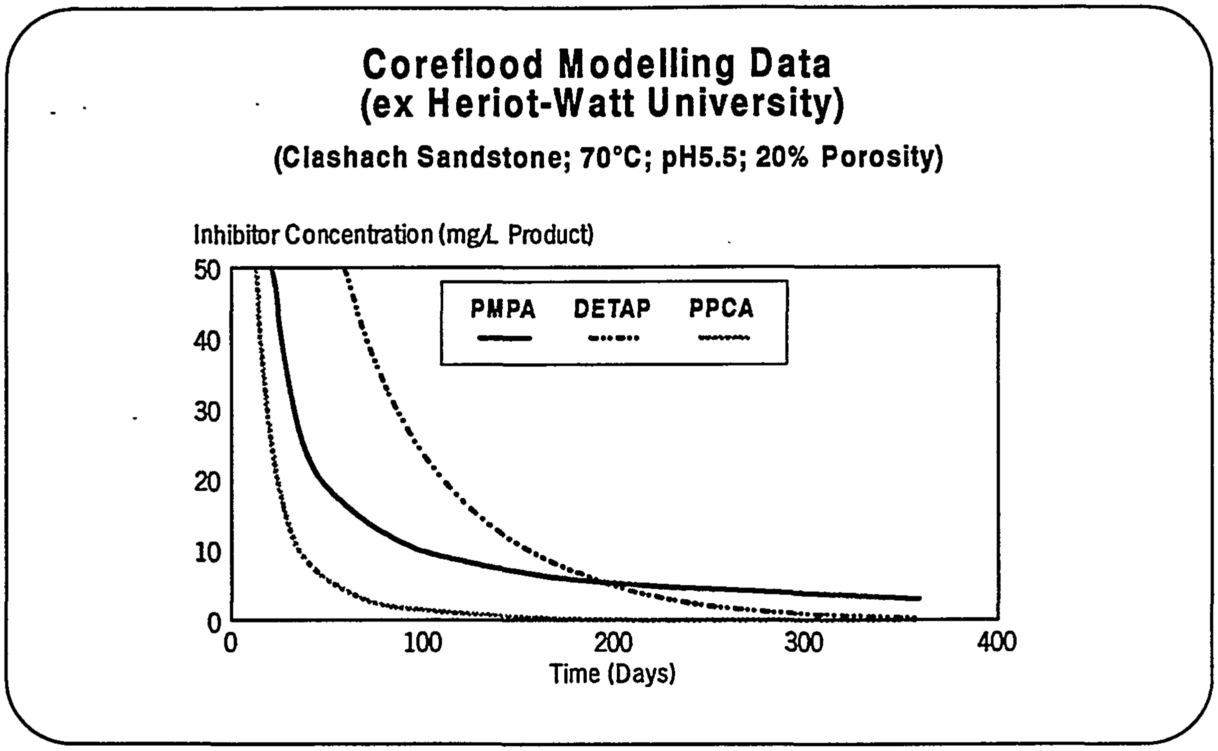


Fig. 3

1. The core-flood and modelling studies carried out by the OSRG at Heriot-Watt University Department of Petroleum Engineering (Fig. 3) demonstrated the highly efficient adsorption/desorption behaviour of a PMPA and predicted that it would desorb at a consistent, sustained concentration in the core-flood returns.

2. The residual level of PMPA in the modelled desorption profile greatly exceeds that of the DETAP, giving in excess of 350 days compared to approximately 200 days for the latter.

3. The PPCA has relatively poor inherent adsorption/desorption properties and these results confirm that it needs a (partial) precipitation technique to enhance its performance.

4. The in-house (Petrolite Ltd) core-flood studies (Fig. 4) confirmed the predicted outstanding desorption profile. Under exactly the same core-flood test conditions to the independent (Heriot-Watt University OSRG) work the PMPA product was still returning at a level of 4 to 5 mg/L after 4,500 pore volumes when the test was terminated due to core-holder failure. The DETAP had reached a residual level of less than 5 mg after approximately 1,500 pore volumes.

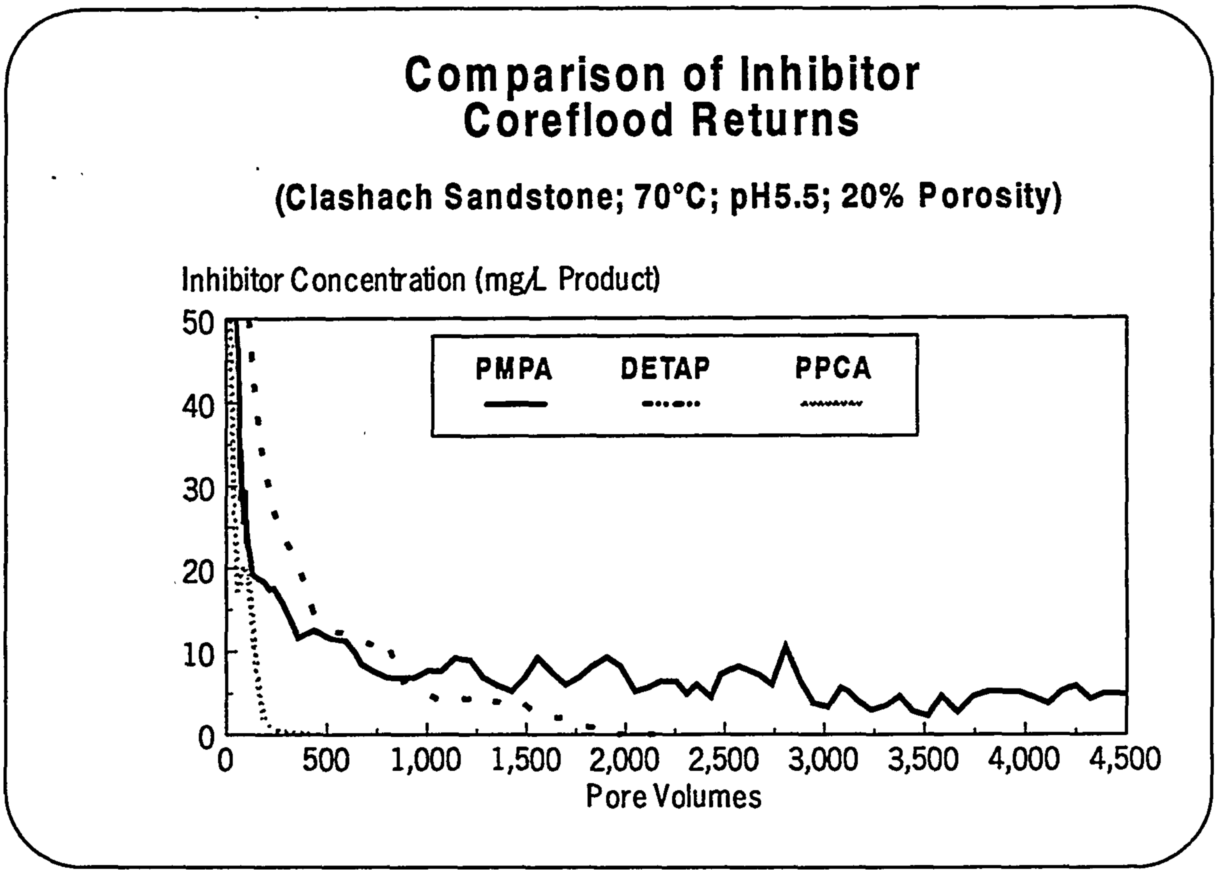

Comparison of Inhibitor Coreflood Returns
(Clashach Sandstone; 70°C; pH5.5; 20% Porosity)
Inhibitor Concentration (mg/L Product)
50
40
30
20
10
0
PMPA
DETAP
PPCA
0
500
1,000
1,500
2,000
2,500
3,000
3,500
4,000
4,500
Pore Volumes

Fig. 4

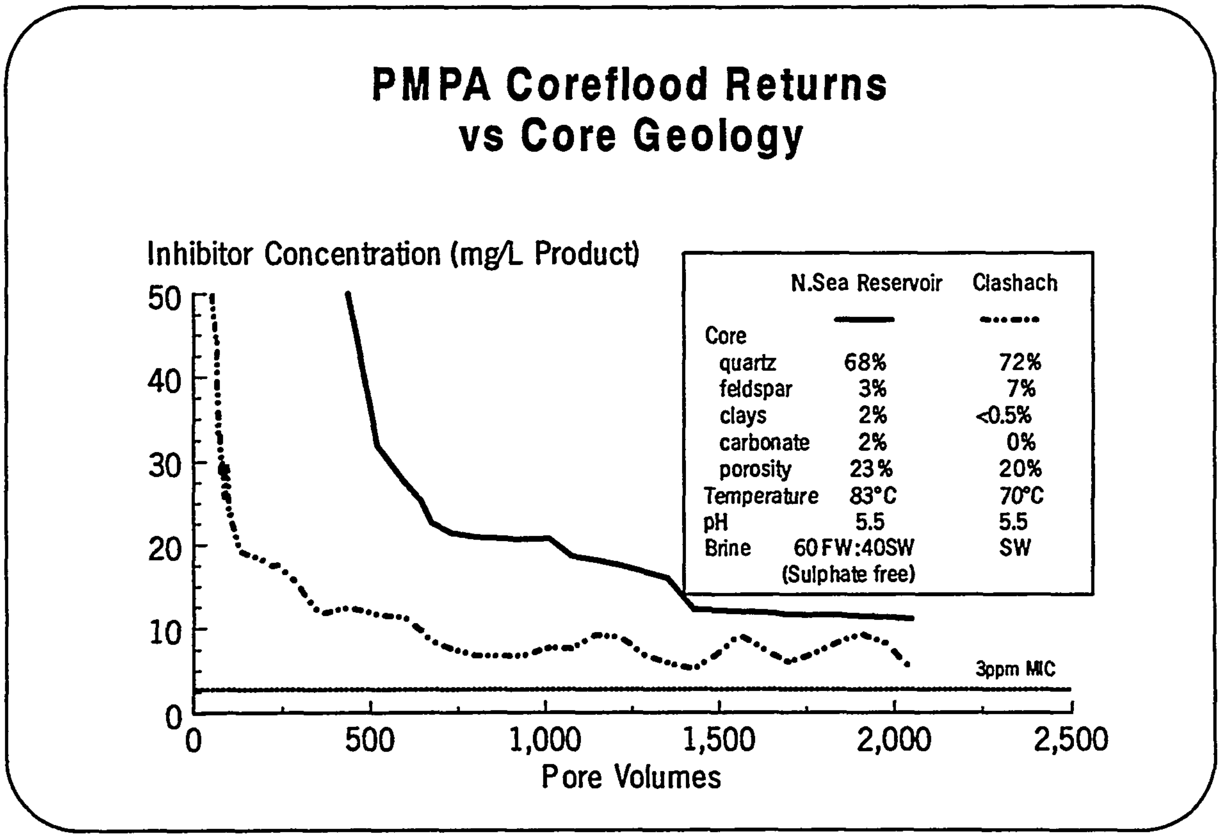

PMPA Coreflood Returns vs Core Geology
Inhibitor Concentration (mg/L Product)
50
40
30
20
10
0
N.Sea Reservoir
Clashach
Core
quartz 68% 72%
feldspar 3% 7%
clays 2% <0.5%
carbonate 2% 0%
porosity 23% 20%
Temperature 83°C 70°C
pH 5.5 5.5
Brine 60FW:40SW (Sulphate free) SW
3ppm MIC
0
500
1,000
1,500
2,000
2,500
Pore Volumes

Fig. 5

5. The adsorption/desorption characteristics of PMPA can significantly improve in the more complex mineralogical conditions of North Sea reservoirs than Clashach sandstone, which is relatively low in "clays".

   This effect of mineralogical composition has been studied in depth, and reported by other workers in this field. [12, 13]

   The effect on the desorption profiles due to the presence of clays and calcite can be demonstrated by the comparison between Clashach core and a field core (Fig. 5). The presence of these minerals can significantly enhance the adsorption dynamics of PMPA and thus provide higher residual levels and a greater treatment capacity.

   This improvement may also be attributed to the higher temperature and the presence of higher levels of calcium existing in the "real case" than in the simulated (sea water) brine used in this test work.

6. The overall modelling conclusions which can be drawn are that PMPA could provide a potential for reducing the volume of product, reducing the concentration of product, reducing the volume of overflush, or even a combination of all three.

Scale Inhibition Performance

During our evaluation of PMPA for squeeze treatments, we needed to ensure that any products developed would successfully prevent mineral scale deposition. Both static (bottle) type precipitation and dynamic (capillary loop blocking) tests using in-house test procedures were used to evaluate the performance of the novel PMPA product.

A summary of test procedures is presented here:-

1. Dynamic (Capillary Loop Blocking) Test

   The Dynamic test consists of two positive displacement pumps which pump brines to a mixing chamber. The mixed brine flows through a thermostatically controlled, heated capillary tube and out to waste. A pressure transducer records the back pressure on a data logger. A pressure switch is incorporated for protecting the apparatus which stops the pumps after a predetermined pressure limit is reached (Fig. 6).

   A two-part brine was prepared. One brine contained the scaling metal cations such as calcium and barium, whilst the other contained the scale forming anions sulphate and bicarbonate. The two brines were pumped at an appropriate rate through the capillary and the back pressure monitored.

Scale depositing in the capillary resulted in a rise in the back-pressure which eventually reached the limit of the pressure switch when the pumps were stopped. At this point the transducer-recorder system and pressure switch were isolated and the capillary flushed with a cleaning solution. Following the generation of a scaling blank as described above, the candidate scale inhibitors were tested at varying dosages to assess their scale inhibiting performance.

For carbonate scales, the cleaning solution used was 15% nitric acid. For sulphate scales, a chelating agent such as EDTA was used. When the pressure drop returned to the original baseline pressure, the capillary was thoroughly flushed with deionised water.

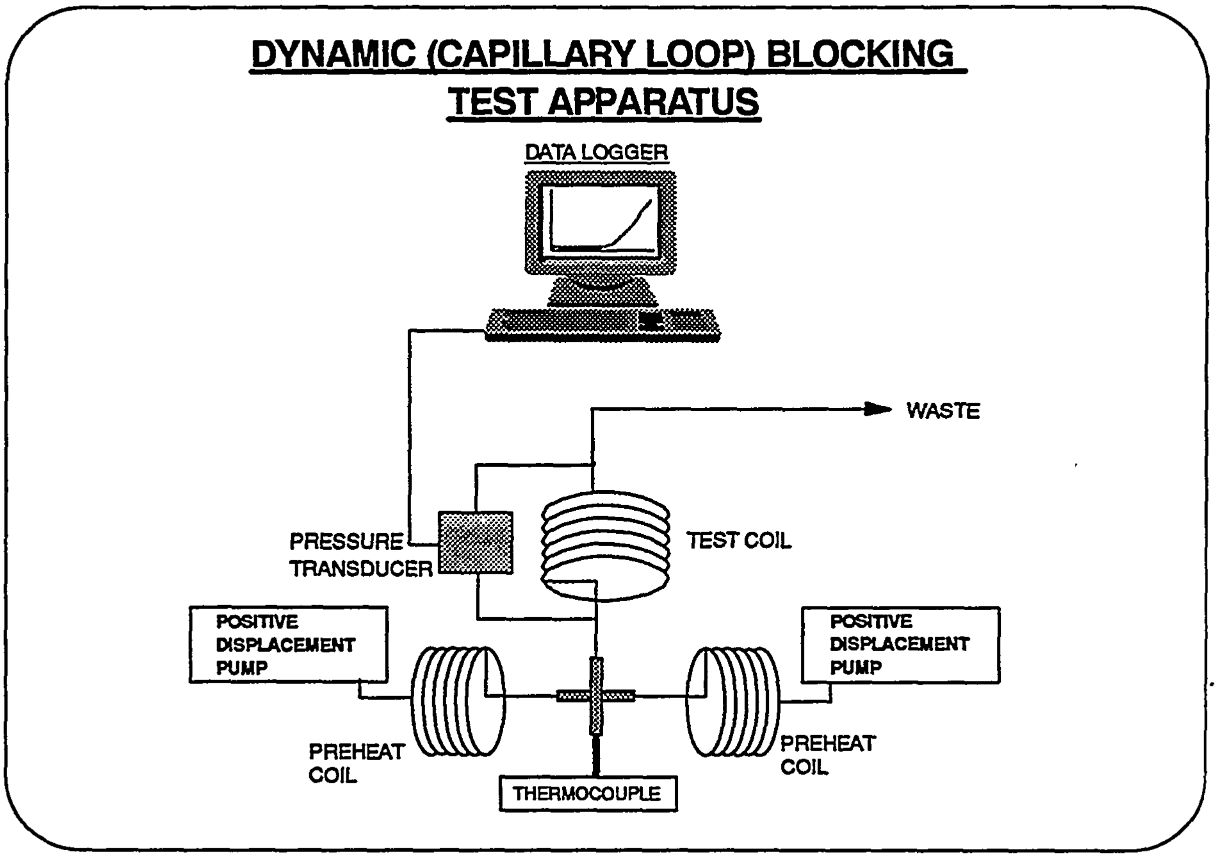


Fig 6

2. Static (Precipitation) Bottle Test

A two part brine based on the relevant water analysis was made up and buffered as necessary to the required pH using a buffering agent, e.g. imidazole for the barium sulphate test. The calcium carbonate test was unbuffered. The scale preventatives to be evaluated were made up as either 1% or 10% solutions in distilled water, depending upon the overall dosage level required.

To a series of 50 ml disposable glass bottles, 25 ml of scaling anionic solution was added using an EDOS electronic dispensing pipette. To one set of three bottles, 25 ml of deionised water was added instead of anionic solution, these bottles later formed non-scaling blank solutions. Using a micropipette the inhibitor solutions were dosed into the anions in the bottles, in triplicate at each of the dosage levels under evaluation. One set of three bottles was left free of inhibitor, these eventually formed "scaling blank" solutions. Both sets of blanks were utilised later to determine the percent inhibition provided by each dosage of inhibitor.

The bottles were then immersed in a water bath, along with the stock cation solution so that both could be raised to system temperature. At system temperature 25 ml of the scaling cationic solution was added to all bottles, again using the EDOS dispenser. The bottles were then sealed and left for the required time interval, after which an aliquot (1 ml) was removed and added to 9 ml of fixing solution. These solutions were then analysed for the required residual scaling cations and subsequently percent inhibition levels determined for each inhibitor dosage.

Percent inhibition levels are determined as follows:-

$$\%\ \text{Inhibition} = \frac{(a - y)}{(b - y)} \times 100$$

a = Average cation concentration in sample

y = Scaled blank (Average cation concentration in anions and cations - no inhibitor present)

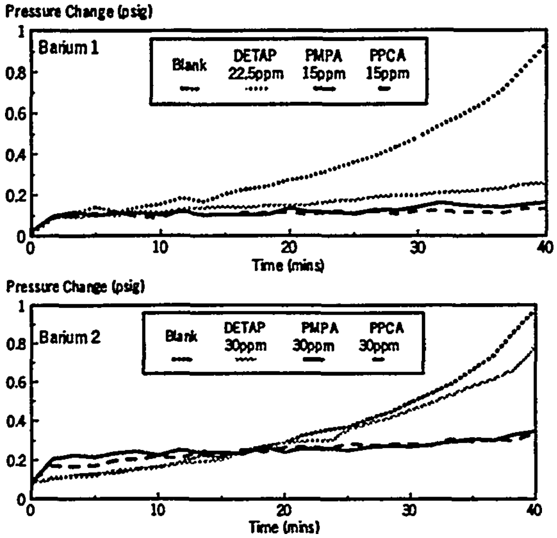
b = Unscaled blank (Average cation concentration in cations and dionised water)

Scale Inhibition Performance - Results & Conclusions

1. Barium Sulphate

Barium Loop Test Data

(Test Temperature 70°C)



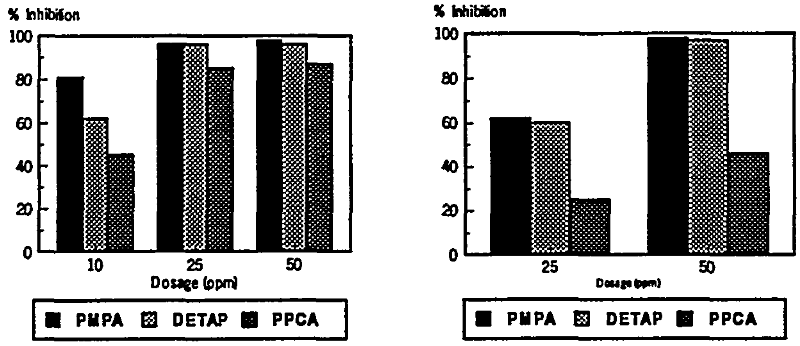
Brine Analysis

	mg/L
Sodium	10,120
Potassium	338
Magnesium	820
Strontium	200
Barium 1	50
Barium 2	100
Sulphate	1,327
Chloride	19,325
TDS	32,180
pH	6
Temperature	70

Fig. 7

Barium Sulphate Bottle Test

(Test Temperature 70°C; pH 6.0; Test Time 2 hours)



Brine Analysis

Ion	mg/L
Barium	50
Sulphate	1327
TDS	30000

Brine Analysis

Ion	mg/L
Barium	100
Sulphate	1327
TDS	30000

Fig. 8

The results of the static bottle test and the dynamic test show some interesting aspects of PMPA performance. Generally in the capillary loop test, which gives an indication of the ability of a scale inhibitor to prevent crystal growth, polymers tend to show better performance. The data from Fig. 7 shows that PMPA has very similar performance to PPCA and significantly better performance than DETAP.
The static bottle test is more of an assessment of the scale inhibitor's ability to prevent scale crystal nucleation and phosphonates e.g. (DETAP) are widely known for this purpose and usually perform better than polymers on this test.

The data from the bottle test (Fig. 8) shows equivalent performance to DETAP but considerably better than PPCA.

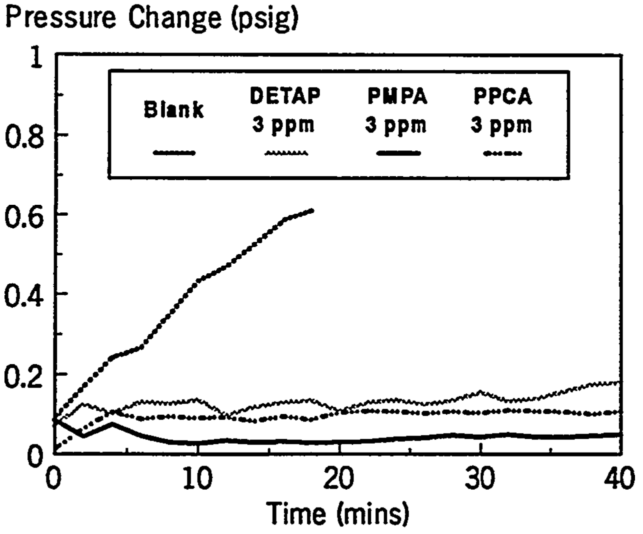
Therefore PMPA combines the benefits of both nucleation and growth inhibition which are shown by each separate DETAP and PPCA product type. This apparent dual mechanistic ability of PMPA to possess phosphonate and polymer attributes adds to the overall efficiency of the product.

2. Calcium Carbonate

The Dynamic (Capillary Loop Blocking) test results show PMPA, DETAP and PPCA have very similar performance at a low (400 mg/L) calcium level (Fig. 9). At a higher (2000 mg/L) calcium level the PMPA lies between DETAP (best) and PPCA (worst) in this test (Fig. 10). The results of the calcium carbonate bottle test (Fig. 11) show that the PMPA is marginally poorer than DETAP and PPCA at very low dosage. However at (more typical) dosages of 5 - 10 ppm there is very little difference in performance.

Loop Test Data (400mg/L Calcium)

(Test Temperature 85°C)



Brine Analysis

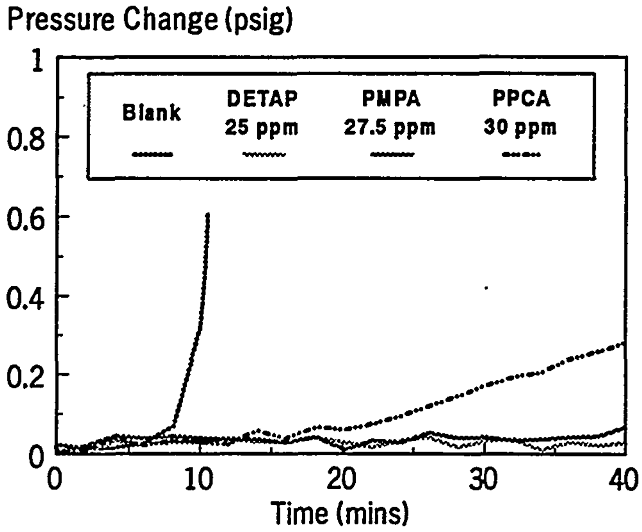
	mg/L
Sodium	9,797
Potassium	338
Magnesium	820
Calcium	400
Chloride	19,325
Bicarbonate	1,500
TDS	32,180

Fig. 9

Overall the PMPA shows acceptable performance against calcium carbonate.

Loop Test Data (2000mg/L Calcium)

(Test Temperature 85°C)



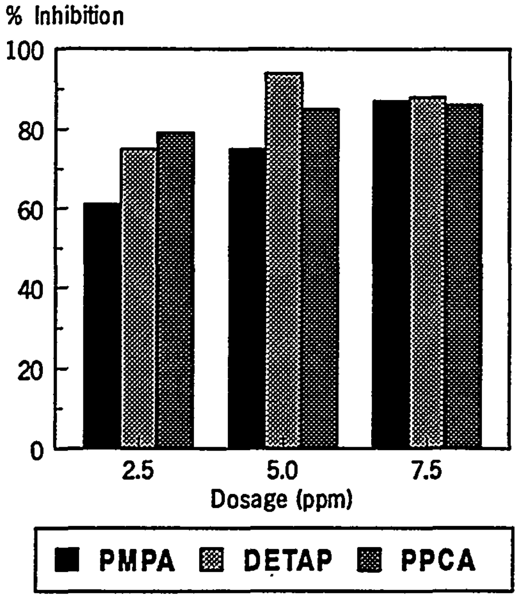
Brine Analysis

	mg/L
Sodium	21,464
Potassium	338
Magnesium	820
Calcium	2,000
Chloride	38,650
Bicarbonate	1,500
TDS	64,772

Fig. 10

Calcium Carbonate Bottle Test

(Test Temperature 85°C; Test Time 24 hours)



Brine Analysis

Ion	mg/L
Calcium	400
Bicarbonate	750
TDS	32180

Fig. 11

Solubility for Squeeze Treatments - Calcium Compatibility

Test work was carried out using synthetic brine systems to simulate the effects of sea water containing the scale inhibitor at varying concentrations mixing with formation water at elevated temperatures. This was essentially a test to assess and compare the compatibility (tolerance) of the inhibitors to calcium at elevated temperature.

A summary of the test procedure is as follows:-

A two part synthetic brine based on the formation water and a synthetic sea water brine were prepared.

Candidate scale inhibitor solutions were prepared at the desired concentrations in sea water. These solutions are usually in the range 1% to 20% but this may vary depending on the amount of calcium in the formation brine.

The scale inhibitor solutions were then mixed at a 50:50 ratio with the synthetic formation brine.

The resultant solutions are then heated to the desired downhole temperature and monitored hourly for the first few hours and then overnight for signs of calcium incompatibility.

The tubes were categorised as follows upon inspection -

1) Clear - the solution in the tube is clear and free from solids.

2) Turbid - the solution in the tube shows opaque colouration indicating formation of fine solids in solution.

3) Precipitate - the solution in the tube has a definite sediment on the bottom. The supernatant may be clear or opaque.

Solubility for Squeeze Treatments - Results & Conclusions

The desirable good solubility DETAP characteristics have been largely duplicated by PMPA, except in some extreme circumstances (Fig. 12). However, in some instances precipitation may be desirable and the tendency to precipitate could be judged as beneficial in some circumstances. This would be very much dependent on shut-in time and hence the true temperature of the brine system under these conditions.

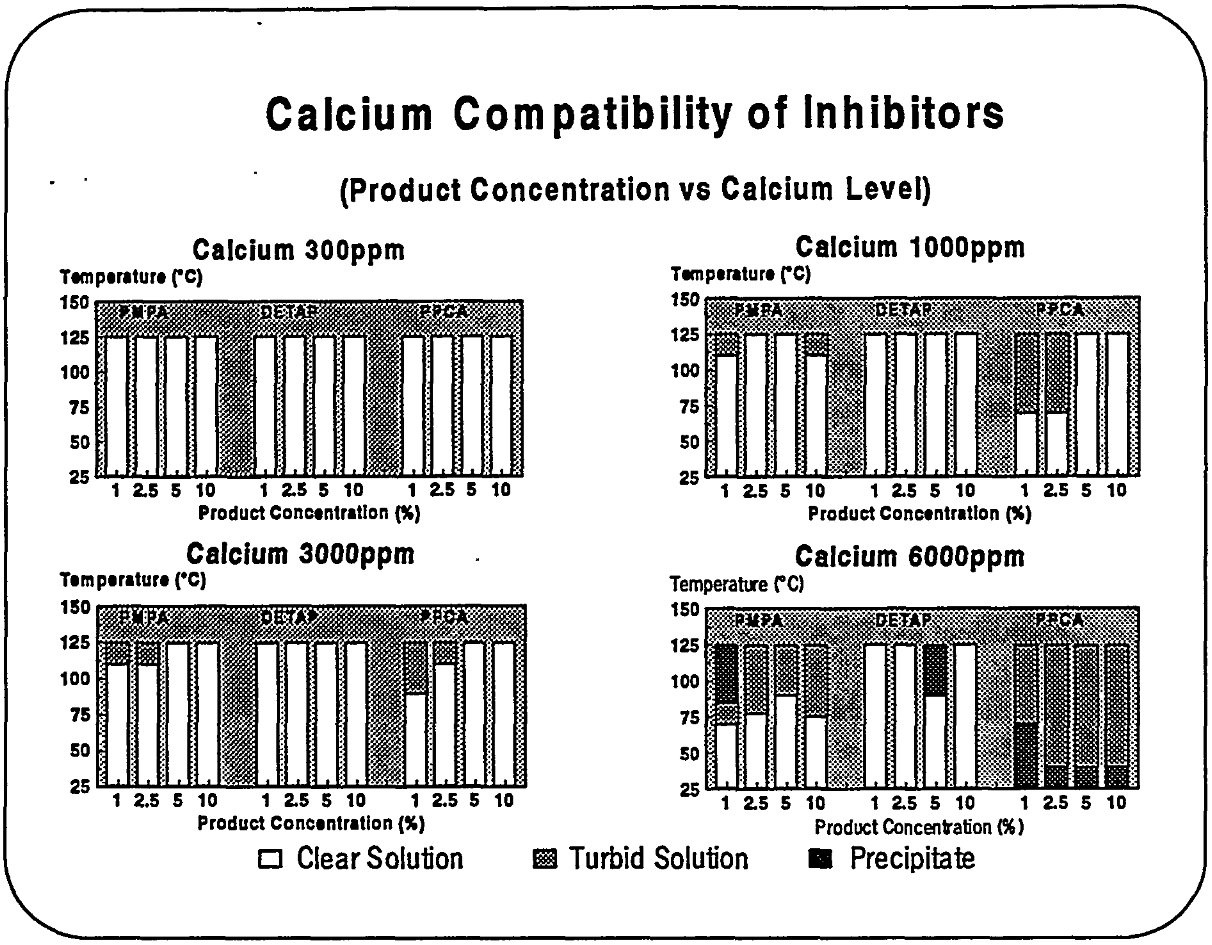


Fig. 12

Thermal Stability

Work is currently in progress to evaluate the thermal stability of PMPA using DETA phosphonate as a control sample.

The main issue, as always, is the development of a suitable test protocol to reflect/simulate field conditions.

At this stage we can state that we would not expect the thermal stability of PMPA to be significantly different from DETA phosphonates given the fact that the former is a phosphonated polyamine.

Indeed some early studies indicate that the thermal stability of PMPA is superior to DETA phosphonates.

ENVIRONMENTAL

Regulatory test data indicates a PMPA to possess relatively low toxicity and moderate biodegradability and is non-bioaccumulative. These facts, coupled with the more efficient desorption behaviour which leads to lower chemical discharge rates, suggest PMPA will have negligible effect on the environment.

MAIN CONCLUSIONS

The work carried out has shown that the novel chemistry of PMPA can provide:-

1. Longer squeeze life in terms of treated cumulative produced water volume compared to current phosphonates.

2. The benefit of a single squeeze component - no extra additives or complicated procedures are required.

3. Potentially significant reduction in treatment chemical volume required for a successful squeeze treatment.

4. Potentially significant reduction in treatment chemical concentration required for a successful squeeze treatment.

5. Significantly improved barium sulphate inhibition compared to DETAP and equivalent to PPCA.

6. Excellent solubility characteristics for squeeze treatment in high TDS, high calcium brines.

There is a great deal of scope within the novel chemistry to utilise different amine backbones and degrees of polymerisation, thus offering scope for a range of products which nominally could be adapted for specific water chemistry/reservoir conditions. This would be within the scope of Petrolite patents on chemistry, manufacture and application.

Overall we believe we have developed a novel product which, with correct application considerations and planning, will give much more efficient squeeze treatments resulting in longer lives, with reduced chemical wastage and hence reduced environmental impact and overall cost to the operator.

ACKNOWLEDGEMENTS

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