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Evaluation of Diethylene Triamine-pentamethylene Phosphonic Acid (DTPMP) as Scale Inhibitor of Calcium Carbonate Scales in Oil Field Water

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ABSTRACT: *Scale formation is one of the most serious oil field problems that inflict water injection systems. This study was conducted to investigate the permeability reduction caused by deposition of scale in oil field water where contained high concentration of calcium ion at various temperatures (40, 60, 80 and 95 °C). The aim of this work is study the prevention or minimizing of scale formation in oil field by using economic and good performance inhibitor. The scale inhibitor phosphonate type (DTPMP) was conducted at low and high temperatures, was shows a good performance at low concentrations 3 to 20 ppm. Static test was carried out to demonstrate the best concentrations of scale inhibitors at different temperatures. The Dynamic test has also done for the sample by using dynamic scale rig test at a temperature of 75°C and injection rate of 2 liter per hour for sample (scaling solution) to determine the efficiency of scale inhibitor Diethylene triamine-pentamethylene phosphonic acid (DTPMP) at injection rates of (3, 5, 7, 10, and 20ppm). From the both static and dynamic experiments we conclude that the performance of scale inhibitor (DTPMP) is effective in preventing calcium carbonate* $CaCO_3$ *scale, where it has achieved efficiency at 40, 60, 80 and 95*^oC *was 100, 98.20, 94.83 and 88.50% respectively, at concentration 10ppm. The performance of scale inhibitor considered an effective inhibitor where the minimum performance was 60.00 – 96.23% at low concentration (3 ppm); this is considered acceptable performance and economic as scale inhibitor in the oil fields water.*

Keywords: *Diethylene triamine-pentamethylene phosphonic acid (DTPMP), Scale inhibitor, static test, dynamic test, jar test, scale rig.*

I. INTRODUCTION

Water is one of the most abundant and important substances on earth. It dissolves almost all inorganic substances to some extent. The solvent power of water is great because of its molecule structure, its ability to act as an acid or a base, and the fact that it can function as either an oxidizing or a reducing agent. Theoretically the angle between the hydrogen bonds to the oxygen is 90^o but because of repulsive forces, it is actually 105°. This results in a polar molecule, with the oxygen end being partially negative and the end with the hydrogen partially positive. Since inorganic compounds are ionic in nature, A tends to dissolve in polar solvents. Pure water is not very corrosive to steel. Corrosion will proceed until the corrosion products stifle the reaction. The addition of salt to water increases conductivity and corrosion. Dissolved gases such as oxygen and carbon dioxide increase the corrosives of the water [1]. In fact; dissolved gases are the primary cause of most corrosion problems. If they could be excluded and the water maintained at a neutral pH or higher, most oilfield waters would cause very few corrosion problems [2]. All types of oilfield equipment exposed to water are subject to corrosion. Corrosion can cause not only a shutdown in production operations, but also causes a safety hazard by weakening high pressure equipment. Leaks in flow lines or pipelines can also result in costly damages to a farmer's property. Corrosion products removed by turbulent water flow can deposit in processing equipment to reduce permeability in water injection wells. Damage caused by corrosion can, therefore, decrease the operational efficiency of a system [1]. The formation of scales and sludges deposited from waters is troublesome. These deposits may form in distribution lines, domestic hot water heaters, various types of cooling equipment, boilers, heat exchangers, or on nearly any surface which water contacts. These deposits often prove expensive due to shutdowns of equipment for removal of deposits or replacement of the equipment. Scale formation in gas and oil wells is a common and persistent problem during production, treatment, transportation, and disposal of co-produced salt water; inhibition of this scale formation is a priority [3]. Scale and sludge are differentiated on the basis that scale is a deposit formed in place on surfaces in contact with water, while the sludge may form in one place and be deposited in another. Sludges may collect in areas of a system where the flow rate is low or where there are bends in the lines, and thus build up a deposit which will reduce the flow.

Scale deposition can occur in any place where a significant temperature differential occurs, such as in well tubular, in pipelines and heat exchangers, it can seriously degrade performance, it can also cause problems at point where significant amount accumulate in separators. It is therefore not a local problem, but may occur anywhere within the production process [4, 5].

In the reservoir the brine is in chemical equilibrium with its surroundings at their temperature and pressure. During production processes the equilibrium is disturbed since the brine is moved to a lower temperature and pressure. Solubility of $CaCO_3$ will decrease as a result of pressure drop, and thereby increase the saturation ratio for $CaCO_3$, while a temperature drop causes the opposite influence. The net effect of a drop in temperature and pressure may therefore be an increase or a decrease in the $CaCO_3$ saturation ratio, depending on the temperature change relative to the pressure change. $CaCO_3$, $FeCO_3$, $CaSO_4$ and $SrSO_4$ behave somewhat untypical compared to most salts as the solubility decreases with increasing temperature. $BaSO_4$ is more complicated as it has a maximum in solubility around 100 °C [4, 6].

There is an important difference in the aqueous chemistry for sulfate scales and carbonate/ sulfides scales. While the sulphates are more or less independent of pH, there is a strong pH dependence on the solubility of carbonates/sulfides. This makes prediction of carbonates/sulfides far more complicated than prediction of sulphates since it is necessary to calculate both pH and the concentrations of all the carbonate/ sulfides species. The presence of other acids, such as organic acids, must also be included in the calculations. One of the main reasons for $CaCO_3$ precipitation during oil recovery is the pH increase due to loss of CO_2 from the aqueous phase to the oil and gas phase as the pressure drops [3, 7, 8,].

Two waters are called incompatible if they interact chemically and precipitate minerals when mixed. A typical example of incompatible waters are sea water with high concentrations of SO_4^{2-} and low concentrations of Ba^{2+}/ Sr^{2+} and formations waters with very low concentrations of SO_4^{2-} but high concentrations of Ca^{2+}, Ba^{2+} and Sr^{2+}. Mixing of these waters may therefore cause precipitation of $CaSO_4$, $BaSO_4$ and/or $SrSO_4$. Sea water is frequently injected into the reservoir to maintain the reservoir pressure and increase the oil recovery [3]. Other fluid incompatibilities include sulfide scale where hydrogen sulfide gas mixes with iron, zinc or lead rich formation waters [5, 9]:

Scale control can be achieved through operation depending on formation of scale. If the scale produced from mixing of two different water, one of them contains high concentration of anions (carbonate ion) and the other contains high concentration of cations (calcium ions), which mostly comes from different reservoirs. In this case the methods of inhibition are separation of the different waters and prevent it from mixing again. While if the formation of scale depending on other factors, the most direct method of inhibiting formation of scale deposits is using the scale inhibitors [4]. The general types of organic materials used as scale inhibitors of calcium carbonate are phosphonates, diphosphonates, phosphate esters, and polyacrylate. Some phosphonate group containing compounds reportedly exhibit greater temperature stability and do not have any harmful degradation products [1]. Most good scale inhibitors should work at dosage levels between 5 and 25 ppm on most production systems depending on calcium concentration [10]. The scale inhibitors not only measured by high effectiveness to inhibit scale formation, but also have good adsorption–desorption characteristics, which determine the operation duration of the scale inhibitors [11]. The treatment rates can be affected by contaminants which take the scale inhibitor out of solution. Some of these contaminants are cationic treating chemicals (corrosion inhibitors, biocides), suspended solids, oil and paraffin. The materials can reduce the concentration of the scale inhibitor in the water phase by chemical adsorption on the surface, or entrainment at the oil / water interface due to surface activity. In these cases more scale inhibitor than normal may be needed to override these compatibility factors and maintains the required scale inhibitor concentration in the water phase [5, 12].

Phosphonates inhibiting crystal growth and scale formation and they are quite stable under high temperature, low and high pH and in the presence of oxidants (harsh chemical conditions). An important industrial use of phosphonates is in cooling waters, desalination systems, and in oil fields to inhibit scale formation [7].

Polyphosphates are among the most strong scale inhibitors, inhibits the crystal growth at concentrations far below those required to chelate stoichiometric amounts of the reactive cations [7,13]. Elayatt, et al, 2016 [10] evaluated six different compounds of phosphonate containing groups, have been synthesized and evaluated for scale inhibition property, the results show that one and two phosphonate group containing compounds do not show any characteristic of prevention of scales of either calcium carbonate or calcium sulphate. The compounds which have three or more phosphonate group show characteristics of prevention of both types of scales. Liu and Nancollas 1973 [14] have reported on their extensive studies of the effect of various phosphonates on the crystal growth of sparingly soluble salts. Pervov 1991 [15] evaluated the performance of various polyphosphates and phosphonates on deposition of gypsum on cellulose acetate and composite membranes at different recovery ratios. The results of his investigation show that compared to phosphonates (i.e., AMP, HEDP), polyphosphates (i.e., sodium tripolyphosphate STPP, and sodium hexametaphosphate SHMP) exhibit superior performance. Amjad 1996 [16] reported that Monsanto has tested various phosphonates**,** and has shown that, for calcium carbonate scale control, the order of effectiveness among various phosphonate is HEDP > AMP > DETMP. Sherbondy and Vanderpool 1995 [17], show that the polyether polyamino methylene phosphonate and corresponding N-oxides of the compositions and methods of the present invention are prepared first by phosphonomethylation of the appropriate primary amine which already contains the polyoxyethylene and polyoxypropylene moieties, followed by an oxidation step which provides the N-oxide moieties. El-Shall et al 2002 [18] studied the effect of Aminotris methylenephosphonic acid [ATMP] on calcium sulfate dehydrate (gypsum) crystallization, and their results indicate that: ATMP increases the induction time at all the supersaturation ratios studied due to decrease the regular crystal growth. Nucleation rate is increased in the presence of ATMP compared with the baseline. The Critical nucleus diameter and hence size is smaller with addition of 100 ppm ATMP. The crystal growth rate is lower with ATMP compared with the baseline. The crystal growth rate is generally lower at lower supersaturation ratio with and without ATMP additive. Amjad et al 2003 [19] , showed that phosphonates and polyphosphates prevent scale formation at "substoichiometric" dosages by adsorbing onto crystal growth sites of micro-crystallites thereby interfering with crystal growth and altering the crystal growth morphology. In addition, both phosphonates and polyphosphates have been shown to exhibit metal chelation and dispersancy activities. Polyacrylic acid, aminotrimethylenephosphonic acid, and polyamino polyether methylenephosphonate was studied as Calcium carbonate scale inhibitors [20]. Temperature and concentration effects of polyphosphates, polyphosphonate and polycarboxylate inhibitors on calcium carbonate precipitation were evaluated and only polyphosphonates remained efficient at high temperatures [21]. In this paper, an inhibitor, which is based on the Diethylene triamine-pentamethylene phosphonic acid (DTPMP), is studied at low and high temperatures.

II. Materials and Methods

2.1. Chemicals Used

All chemicals used is reagent grade, were purchased from BDH (UK). UHQ water was used throughout this study. All experiments were conducted at least in duplicate, and the average of the results reported. Variation in the experimental results is presented as average ± standard deviation of the mean values. The scale inhibitor used in this study is Diethylene triamine-pentamethylene phosphonic acid (DTPMP) syntheses as method used in paper by Elayatt et al 2016 [10]. The chemical structure of (DTPMP) is shown in figure 1.

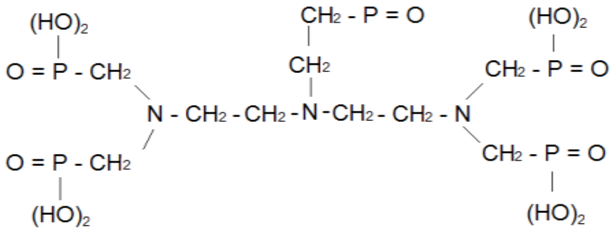

Figure1: Chemical structure of diethylene triamine-pentamethylene phosphonic acid (DTPMP)

2.2. Static test

This method is manly based on the API (American Petroleum Institute) static jar test the main difference being that it evaluates the inhibition of carbonate and sulphate scales together rather than separately. This means that testing can be carried out on single brine containing both carbonate and sulphate ions instead of each single brine.

This adaptation allows the screening procedure to be speeded up especially where a large number of scale inhibitors are to be screened. In this method, the cations and anions of formation water, which taken from producing wells, will determined, these analysis entered to the computer program sheets (Oddo and Tommson), which it calculates the amount of cations and anions for each sample, and then use this brine in the test procedure for the evaluation of scale inhibitors by determined the calcium ion before and after the precipitation for the blank (without any inhibitor addition), to calculate the efficiency of scale inhibitor at different temperature and different dosage rate, the samples will be stay in oven 24 hrs for precipitation at required temperature and dosage[22].

2.3. Sample Preparations

Two solutions are made up. One contains the anions and the other containing the cations. These two solutions are then heated separately to the required temperature before mixing. This procedure ensures that no scaling occurs before the start of testing. In highly scaling brines, it is quite possible to find that major scaling has occurred by the time that they have been transported to the lab and this can lead to erroneous results.

Most scale inhibitors work by modifying or preventing crystal growth and so are only effective if introduced to the system before any scale is formed.

Analysis of the brine is carried out and the concentration of cations and anions are determined for formation water taken from Waha oil field, Libya, well Q-25.These concentrations for cations and anions are carried into the Oddo and Tommson program which calculate the amount of the synthetic brines.

The Cations and anions as shown in the table (1) above were weighted and transferred separately in a 1000ml calibrated volumetric flask. The flask was then filled up using deionised water and adjust the pH to that the system for sample well Q-25, and deionised water was added drop wise using a pastel pipette to bring the level to the mark.

All two solutions (Cations & Anions) were filtered through a 0.45 micro membrane filter before used and each of solutions (Cations & Anions) were transferred into a separate Pyrex dispenser bottle and cap, and invert the bottles as is necessary to dissolve the solids, and to make homogenous solution. The bottles were placed in an oven set at the required test temperature (40, 60, 80 and 95°C).

Table1: Synthetic Brine

Synthetic Brine Composition.				
Volume of brain required 1000 ML	Chemical Purity	Chemical Required g/l	g Chemical Required For x ml of Solution	g Chemical Required For x ml of Separate Solutions
CATIONS				
Sodium Chloride Na	0.999	68.0226	68.0226	136.0452
Sodium Chloride Cl	0.999	68.0451	68.0451	136.0903
Potassium Chloride	0.995	0.0000	0.0000	0.0000
Calcium Chloride $2H_2O$	0.995	22.7763	22.7763	45.5526
Magnesium Chloride $6H_2O$	0.980	9.3755	9.3755	18.7510
Barium Chloride $2H_2O$	0.990	0.0000	0.0000	0.0000
Strontium Chloride $6H_2O$	0.985	0.0000	0.0000	0.0000
Ferric Chloride soln.	0.414	0.0493	0.0493	0.0986
ANIONS				
Sodium Sulphate $10H_2O$	0.990	2.0346	2.0346	4.0691
Sodium Carbonate $10H_2O$	0.990	0.0000	0.0000	0.0000
Sodium Bicarbonate anhyd	0.995	0.3875	0.3875	0.7751
Sodium Sulphide anhyd	0.995	0.0000	0.0000	0.0000

Prepare the required number of new clean 100 ml wide neck screw cap bottles (12 bottles for each sample) and mark them up with the scale inhibitor (DTPMP) being tested and the dose rate (3, 5, 7, 10 and 20ppm). The test should be carried out in duplicate with two blanks i.e. containing no chemical. Remove the bottle of cation solution from the oven and fit a clean dry repeat dispenser. Set the dispenser to 50 ml and prime it to remove any air from the pump mechanism and Dispense 50 ml of cation solution into each of the 100 ml test bottles. Set the two blanks to one side.

Using a micro syringe 50µl, add the required volume of 10% scale inhibitor solution to each bottle (3, 5, 7, 10 and 20ppm), cap all bottles and shake to mix. Remove the anion solution from the oven and fit a clean dry repeat dispenser. Set the dispenser to 50 ml and prime it to remove any air from the pump mechanism and Dispense 50 ml of anion solution into each of the 100 ml test bottles, cap each of the bottles and shake to mix. Note the time, inspect each of the bottles for signs of scaling or scale inhibitor, and note all observations on the test sheet, after that immediately place all the test bottles in the oven. Repeat the observation procedure at 1, 2, 4, 8 and 24 hours and note all observations.

1- Carbonate scale will tend to form a "scum" on the surface of the liquid.
2- Sulphate scale will tend to form a crystalline layer on the bottom of the bottle.
3- Incompatibility and precipitation of the scale inhibitor will tend to show as a fairly even haze throughout the liquid. At the end of the 24 hour test period, allow the bottles to cool. Remove an aliquot from each bottle and filter it through a 0.4 micron Millipore filter. Analyze immediately for calcium on each filtered solution and record all results on the observation sheet. From the equation below, the calcium concentration in the sample ($CaCO_3$) can be calculated as:

$$\mathbf{Ca^{++}\ as\ CaCo_3(ppm)} = \frac{Eq.wt(CaCo_3)\times(0.02N)EDTA\times TR\times 1000}{Sample\ Volume}\times \mathbf{0.4}$$

Where:
TR = Titration Reading
N = Normality of EDTA solution = 0.02 N
Volume of sample of Burette (ml)
Eq.Wt for $CaCO_3$ = 50 g/mol

Multiply all the original calcium test results obtain as $CaCO_3$ by 0.4 to give calcium ion (Ca^{++}) in the sample and record all calcium readings as (mg/l Ca^{++} Sample) in the table above.

$\mathbf{C_a = Ca^{++}}$ concentration

We have used the calcium ion from the static jar test to calculate the percent inhibition by using the following equation:

$$\% \text{ Inhibitor} = \frac{C_a - C_b}{C_c - C_b} \times \mathbf{100}$$

Where: in the treated sample after precipitation
$\mathbf{C_b = Ca^{++}}$ concentration in the blank after precipitation
$\mathbf{C_c = Ca^{++}}$ concentration in the blank before precipitation

2.4. Evaluations of scale inhibitors (Dynamic Test) using the P-MAC (Pressure Measurement and Control) Dynamic Scale Rig.

2.4.1. Principle of Operation

This equipment consists of a micro-bore tube housed in a temperature controlled fan oven as shown in figure 2. Separate anion and cation solutions are first pumped through a mixing tube before entering the micro-bore scaling tube. This is achieved by means of a speed controllable peristaltic pump. When the two mixed solutions enter the scaling tube, scale is formed and adheres to the walls causing the bore to be reduced and a pressure increase. The pressure differential across the scaling tube is measured by means of a pressure transducer. The electronics then process this signal to give an output to the chart recorder. The required dose rate of scale inhibitor can be introduced into the scaling solutions by means of a second speed controllable peristaltic pump feeding into a T piece at the entrance to the scaling tube.

2.4.2. Procedure

After flushing, immerse one tube from Pump (A) in Solution (A) scaling solution and the other tube in Solution (B) scaling solution. Check that the oven temperature remains steady at 75°C, and no air bubbles are formed and then press the “Reset” button on the P-MAC Unit to set pump output. After a short time, an increase in pressure will be observed on the chart recorder graph indicating scale build up within the test coil. Allow the pressure to build up to 10 psi. This will give a blank curve for the scaling solutions being used. (i.e. without any scale inhibition).When the test is complete, flush the system with distilled water and clean out the test coil by disconnecting at the rear of the oven and flushing through with 5% Citric Acid solution and (2M Nitric Acid or 0.1N HCl solutions) using a 50 ml. syringe. Flush out with distilled water and re-connect back into the system. Flush out the system with distilled water to remove all traces of acid and stabilize the oven temperature before starting the next test. After that flushing, immerse one tube from Pump (A) in Solution (A) scaling solution and the other tube in Solution (B) scaling solution. Check that the oven temperature remains steady at 75°C, and no air bubbles are formed and then press the “Reset” button on the P-MAC Unit to set pump output. After a short time, an increase in pressure will be observed on the chart recorder graph indicating scale build up within the test coil. When the observed pressure reach approximately 2.0 - 2.5 psi. Start injection 3ppm of scale inhibitor (A) by Pump (B) [scale inhibitor (A) pump] and turn the three way tap on the inlet tube to the ON position. After a few minutes a leveling off of the scaling curve should be observed on the chart recorder. The speed at which this occurs will depend on the efficiency and dose rate of the scale inhibitor. Repeat the procedure for scale inhibitor (A) at each of the required dose rates (5, 7, 10 and 20ppm).

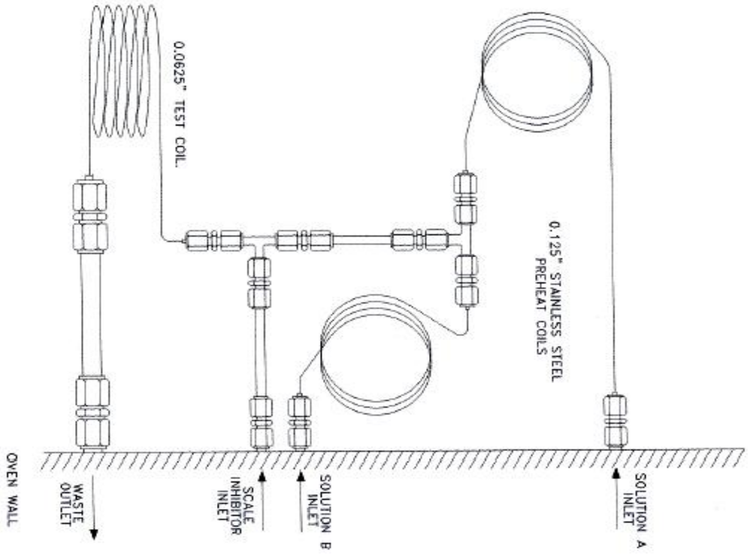

Figure 2: Dynamic Scale Rig Oven Piping Arrangement

III. Results and Discussions

3.1. Analysis of formation water from well No. Q-25

Table 2 shows the analysis of formation water collected from the well No Q25. The analysis contains the analysis of cations (Na^+, Ca^{++}, Mg^{++}, Ba^{++}, Fe total), and the anions (Cl^-, $HCO3^-$, $CO3^{--}$, $SO4^{--}$, S^{--}) , also measuring the specific gravity, temperature, pressure, pH, and the dissolved gases (O_2, H_2S, CO_2) .These data analysis were entered to the program (Oddo and Tommson),which then the program calculate the total ionic strength , calculation pH, and scaling index (Is) at vessel temperature.

Table 2: Ionic strength Composition of Well No.Q-25

Cations			**Anions**		
Ion	**Concentration**		**Ion**	**Concentration**	
	mg/l	**meq/l**		**mg/l**	**meq/l**
Na^+	26741	1163	Cl^-	54810	1546
Ca^{++}	6200	309	HCO_3^-	280	5
Mg^{++}	1096	90	CO_3^{--}	0.00	0.00
Ba^{++}	-	-	SO_4^{--}	600	12
Iron(total)	9	0.332	S^{--}	0.004	0.00
Total moles/ liter		3.0	Total ionic strength		1.77
Specific gravity at 20/20 ^{0}C		1.066	T.D.S (total diss. solids)		89736
Temperature ^{0}C		51	O_2 (mg/l)		0.00
Pressure (psig).		165	H_2S (mg/l)		0.04
pH at 25 ^{0}C & 1 atm.. pressure		6.5	CO_2 (mg/l)		310
Calc. pH at vessel temp.		6.69	Scaling index (Is) at vessel Temp.		0.658

Where: **meq/l** is calculated by dividing the (ppm) of a component by its equivalent weight

3.2. Static jar test results

3.2.1. Static Jar Test results for well No. Q-25

The program (Oddo and Tommson) can also calculates the relationship between the temperature and calcium carbonate formation as shown at Table 3.

Table 3: Calcium Carbonate Scaling Prediction Data

Calcium Carbonate Scaling Prediction Data				
For Pressure165 (Psig)	**Temp ^{O}C.**	**pH**	**Is**	**$CaCO_3$ (mg/l)**
$CaCO_3$ (Scale UNLIKELY)	20	6.46	0.163	-207
$CaCO_3$ (Scale LIKELY)	40	6.61	0.368	259
$CaCO_3$ (Scale LIKELY)	60	6.76	0.888	424
$CaCO_3$ (Scale LIKELY)	80	6.91	1.396	454
$CaCO_3$ (Scale LIKELY)	100	7.06	1.894	459

Table 3 shows that the calcium carbonate will precipitate at 40, 60, 80 and 100°C and no scale formed at 20°C. Figure (4) shows the relationship between the amount of calcium carbonate scale precipitate (mg/l) and temperature where the precipitation of calcium carbonate increases with increasing the temperature.
The program also calculates the relationship between the temperature and calcium sulphate formation as shown in table 4.

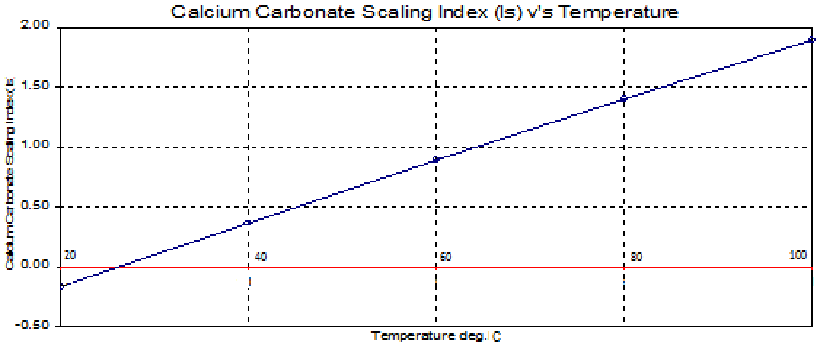

Figure 3: calcium carbonate scaling index (Is) vise the temperature**.**

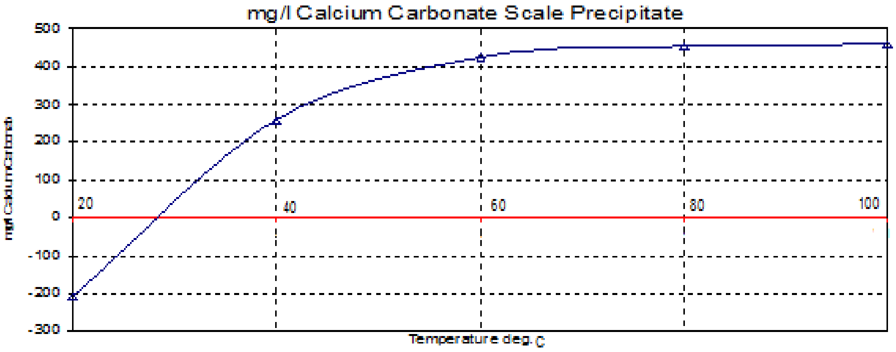

Figure 4: calcium carbonate scale precipitate vise temperature.

Table 4: Calcium Sulphate Scaling Prediction Data

Calcium Sulphate Scaling Prediction Data		
Scaling Tendency at (165) (psig)	**Temp ^{O}C**	**mg/l $CaSO_4$**
CaSO4 (Scale UNLIKELY)	20	-17510.714
CaSO4 (Scale UNLIKELY)	40	-17450.606
CaSO4 (Scale UNLIKELY)	60	-17443.232
CaSO4 (Scale UNLIKELY)	80	-17446.343
CaSO4 (Scale UNLIKELY)	100	-17446.343

Table 5: Magnesium Sulphate Scaling Prediction Data

Magnesium Sulphate Ion Pairs					
Temperature °C	**20**	**40**	**60**	**80**	**100**
Mg^{2+} mg/l	7.02	9.10	10.30	11.35	12.45
SO_4^{2-} mg/l	27.76	35.95	40.70	44.87	49.20
$MgSO_4$ mg/l	34.79	45.05	50.99	56.22	61.65

Table 6: Availability of Sulphate Scaling Prediction Data

Available SO_4^{--}					
Temperature °C	**20**	**40**	**60**	**80**	**100**
After $MgSO_4$	572.24	564.05	559.30	555.13	550.80
After $BaSO_4$	572.24	564.05	559.30	555.13	550.80
After $SrSO_4$	572.24	564.05	559.30	555.13	550.80
After $CaSO_4$	572.24	564.05	559.30	555.13	550.80

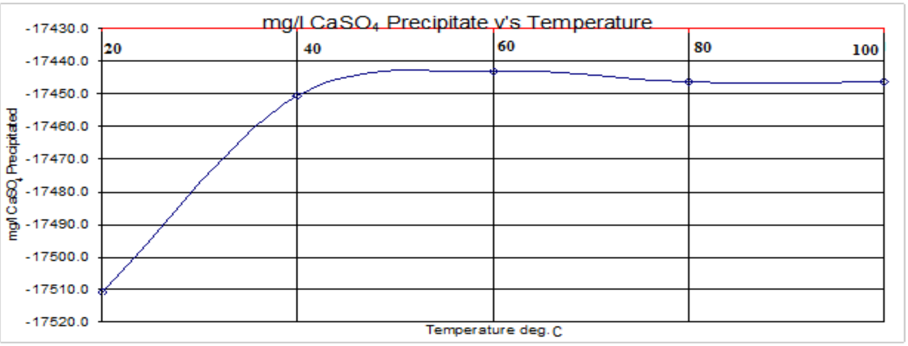

Figure 5: CaSO$_4$ precipitate vise temperature

Tables (4, 5 and 6) and Figure 5 shows there are no calcium sulphate scales formation.

Table 7 shows the observation test for the evaluation of scale inhibitor (DTPMP) with different dosage rate (3, 5, 7, 10, and 20ppm) for 24Hrs period at 40°C. From the observation, the blank shows slight haze after 8Hrs and the precipitation of carbonate becomes clear after 24Hrs. In the other bottles where the inhibitor was injected at different concentrations it observed that all bottles were clear and bright. It can conclude that the scale inhibitor gave very good results with all concentrations.

Table 7: Scale Test Observations at 40^{o}C (Well No. Q-25)

Inhibitor	**Dose (ppm)**	**0 hr**	**1 hr**	**2 hrs**	**4 hrs**	**8hrs**	**24 hrs**
Blank	0	C&B	C&B	C&B	C&B	sl haze	CO$_3$ ppt
(DTPMP)	3	C&B	C&B	C&B	C&B	C&B	C&B
	5	C&B	C&B	C&B	C&B	C&B	C&B
	7	C&B	C&B	C&B	C&B	C&B	C&B
	10	C&B	C&B	C&B	C&B	C&B	C&B
	20	C&B	C&B	C&B	C&B	C&B	C&B
Blank	0	C&B	C&B	C&B	C&B	sl haze	CO$_3$ ppt
(DTPMP)	3	C&B	C&B	C&B	C&B	C&B	C&B
	5	C&B	C&B	C&B	C&B	C&B	C&B
	7	C&B	C&B	C&B	C&B	C&B	C&B
	10	C&B	C&B	C&B	C&B	C&B	C&B
	20	C&B	C&B	C&B	C&B	C&B	C&B

C&B = Clear and bright
sl haze = Slight haze
sl CO$_3$ ppt = Slight Carbonate Precipitate
CO$_3$ ppt = Moderate Carbonate Precipitate

hvy SO$_4$ ppt = Heavy Sulphate Precipitate
SO$_4$ ppt = Moderate Sulphate Precipitate
sl SO$_4$ ppt = Slight Sulphate Precipitate
hvy CO$_3$ ppt = Heavy Carbonate Precipitate

Table 8: Scale test analysis at 40^{o}C for CaCO$_3$ (Well No. Q-25)

Product	Dose (ppm)	Sample 1			Sample 2			Average % inhibition
		mg/l Ca Sample	mg/l Ca Sample -mg/l Ca Blank (C_1)	Percent Inhib. %	mg/l Ca Sample	mg/l Ca Sample -mg/l Ca Blank (C_1)	Percent Inhib. %	
Blank	**0.00**	**5556**	**0.00**	**0.00**	**5556**	**0.00**	**0.00**	**0.00**
DTPMP	3	6118	562	96.23	6119	563	96.40	96.32
	5	6124	568	97.26	6123	567	97.10	97.18
	7	6124	578	98.9	6136	580	99.32	99.11
	10	6140	584	100.0	6139	583	99.83	99.92
	20	6140	584.0	100.0	6140	584	100.00	100.00

From the results obtained from Well No. Q-25 in the table 8 it is observed that the efficiency of inhibition increase by increasing the inhibitor concentration.

Table 9: Scale Test Observations at 60 ^{o}C well (No. Q-25)

Product	**Dose (ppm)**	**0 hr**	**1 hr**	**2 hrs**	**4 hrs**	**8hrs**	**24 hrs**
Blank	0	C&B	C&B	C&B	sl haze	CO_3 ppt	CO_3 ppt
(DTPMP)	3	C&B	C&B	C&B	C&B	sl haze	sl haze
	5	C&B	C&B	C&B	C&B	C&B	sl haze
	7	C&B	C&B	C&B	C&B	C&B	C&B
	10	C&B	C&B	C&B	C&B	C&B	C&B
	20	C&B	C&B	C&B	C&B	C&B	C&B
Blank	0	C&B	C&B	C&B	sl haze	CO_3 ppt	CO_3 ppt
(DTPMP)	3	C&B	C&B	C&B	C&B	sl haze	sl haze
	5	C&B	C&B	C&B	C&B	C&B	sl haze
	7	C&B	C&B	C&B	C&B	C&B	C&B
	10	C&B	C&B	C&B	C&B	C&B	C&B
	20	C&B	C&B	C&B	C&B	C&B	C&B

Table 9 shows the results observation test for the evaluation of scale inhibitor (DTPMP) with different dosage rate (3, 5, 7, 10 and 20ppm) for 24Hrs period at 60°C in the oven. From the observation, the bottles of the blank start slight haze after 4Hrs and the precipitation of carbonate becomes after 8Hrs. In the other bottles where the inhibitor was injected at different concentrations it observed that the 3ppm dosage rate starts slight haze after 8Hrs and in the 5ppm dosage rate the slight haze starts after 24Hrs and the other bottles where the dosages rate 7, 10 and 20ppm were clear and bright. It can conclude that the scale inhibitor gave very good results with 7, 10 and 20ppm.

Table 10: Scale test analysis at 60^{o}C for $CaCO_3$ (Well No. Q-25)

Product	Dose (ppm)	Sample 1			Sample 2			Average % inhibition
		mg/l Ca Sample	mg/l Ca Sample -mg/l Ca Blank (C_1)	Percent Inhib. %	mg/l Ca Sample	mg/l Ca Sample -mg/l Ca Blank (C_1)	Percent Inhib. %	
Blank	**0.00**	**4550**	**0.00**	**0.00**	**4550**	**0.00**	**0.00**	**0.00**
DTPMP	3	5877	1327	83.46	5878	1328	83.35	83.40
	5	5948	1398	87.92	5949	1399	87.99	87.96
	7	6103	1553	97.67	6106	1556	97.86	97.77
	10	6112	1562	98.2	6112	1562	98.24	98.22
	20	6127	1577.0	99.2	6128	1578	99.25	99.23

Table 10 shows the results obtained from Well No. Q-25 at temperature 60 ^{o}C in the table above it is observed that the percent inhibition increase by increasing the amount of scale inhibitor (DTPMP). The best inhibition shows at 7 ppm 97.77% where economically wise best than the other concentrations.

Table 11: Scale Test Observations at 80^{o}C (Well No. Q-25)

Product	**Dose (ppm)**	**0 hr**	**1 hr**	**2 hrs**	**4 hrs**	**8hrs**	**24 hrs**
Blank	0	C&B	C&B	sl haze	sl CO_3 ppt	CO_3 ppt	hvy CO_3 ppt
(DTPMP)	3	C&B	C&B	C&B	sl haze	sl CO_3 ppt	CO_3 ppt
	5	C&B	C&B	C&B	C&B	sl haze	sl haze
	7	C&B	C&B	C&B	C&B	C&B	sl haze
	10	C&B	C&B	C&B	C&B	C&B	C&B
	20	C&B	C&B	C&B	C&B	C&B	C&B
Blank	0	C&B	C&B	sl haze	sl CO_3 ppt	CO_3 ppt	hvy CO_3 ppt
(DTPMP)	3	C&B	C&B	C&B	sl haze	sl CO3 ppt	CO_3 ppt
	5	C&B	C&B	C&B	C&B	sl haze	sl haze
	7	C&B	C&B	C&B	C&B	C&B	sl haze
	10	C&B	C&B	C&B	C&B	C&B	C&B
	20	C&B	C&B	C&B	C&B	C&B	C&B

From table (11) it is clear that the bottles of the blank start slight haze after 2Hrs and the precipitation of carbonate becomes after 4Hrs. In the other bottles where the inhibitor was injected in different concentrations it observed that the 3ppm dosage rate starts slight haze after 4Hrs, the precipitation of carbonate becomes after 8Hrs. And in the 5ppm dosage rate the slight haze starts after 8Hrs and the 7ppm starts slight haze after 24Hrs. The dosages rate 10ppm and 20ppm showed clear and bright. It can conclude that the scale inhibitor gave very good results with 10ppm and 20ppm.

TABLE 12: SCALE TEST ANALYSIS AT 80^{o}C FOR CACO$_3$ (WELL NO. Q-25)

Product	Dose (ppm)	Sample 1			Sample 2			Average % inhibition
		mg/l Ca Sample	mg/l Ca Sample -mg/l Ca Blank (C_1)	% Inhib.	mg/l Ca Sample	mg/l Ca Sample -mg/l Ca Blank (C_1)	% Inhib.	
Blank	**0.00**	**3140**	**0.00**	**0.00**	**3140**	**0.00**	**0.00**	**0.00**
DTPMP	3	5375	2235	75.50	5376	2230	75.66	75.58
	5	5578	2438	81.26	5578	2432	81.10	81.18
	7	5781	2641	88.00	5784	2638	87.93	87.97
	10	5985	2845	94.83	5987	2841	94.70	94.77
	20	6105	2965.0	98.8	5376	2230	75.66	98.80

Table 12 shows the results obtained from Well No. Q-25 in the table above it is observed that the 20 ppm dose rate of scale inhibitor gave a very good result at 80^{o}C.

Table 13: Scale Test Observations at 95^{o}C (Well No. Q-25)

Product	**Dose (ppm)**	**0 hr**	**1 hr**	**2 hrs**	**4 hrs**	**8hrs**	**24 hrs**
Blank	0	C&B	sl haze	sl CO$_3$ ppt	CO$_3$ ppt	CO$_3$ ppt	hvy CO$_3$ ppt
(DTPMP)	3	C&B	C&B	sl haze	sl CO$_3$ ppt	CO$_3$ ppt	CO$_3$ ppt
	5	C&B	C&B	C&B	sl haze	sl CO$_3$ ppt	CO$_3$ ppt
	7	C&B	C&B	C&B	C&B	sl haze	sl CO$_3$ ppt
	10	C&B	C&B	C&B	C&B	C&B	sl haze
	20	C&B	C&B	C&B	C&B	C&B	C&B
Blank	0	C&B	sl haze	sl CO$_3$ ppt	CO$_3$ ppt	CO$_3$ ppt	hvy CO$_3$ ppt
(DTPMP)	3	C&B	C&B	sl haze	sl CO$_3$ ppt	CO$_3$ ppt	CO$_3$ ppt
	5	C&B	C&B	C&B	sl haze	sl CO$_3$ ppt	CO$_3$ ppt
	7	C&B	C&B	C&B	C&B	sl haze	sl CO$_3$ ppt
	10	C&B	C&B	C&B	C&B	C&B	sl haze
	20	C&B	C&B	C&B	C&B	C&B	C&B

Table 14: Scale test analysis at 95^{o}C for CaCO$_3$ (Well No. Q-25)

Product	Dose (ppm)	Sample 1			Sample 2			Average % inhibition
		mg/l Ca Sample	mg/l Ca Sample -mg/l Ca Blank (C_1)	% Inhib.	mg/l Ca Sample	mg/l Ca Sample -mg/l Ca Blank (C_1)	% Inhib.	
Blank	**0.00**	**2840**	**0.00**	**0.00**	**2840**	**0.00**	**0.00**	**0.00**
DTPMP	3	4800	1960	60.00	4800	1960	60.00	60.00
	5	5200	2360	71.50	5208	2368	71.76	71.63
	7	5400	2560	77.50	5401	2561	77.61	77.56
	10	5760	2920	88.50	5758	2918	88.42	88.46
	20	5920	3080	93.30	5924	3084	93.45	93.38

Table 13 shows the results observation test for the evaluation of scale inhibitor (DTPMP) with different dosage rate (3, 5, 7, 10, and 20 ppm) for 24Hrs period at 95°C, where observed that the bottles of the blank start slight haze after 2Hrs and the precipitation of carbonate becomes after 4Hrs. It observed that the 3ppm dosage rate starts slight haze after 4Hrs, the precipitation of carbonate occur after 8Hrs. And in the 5ppm dosage rate the slight haze starts after 8Hrs; at 7ppm starts slight haze after 24Hrs. At 10ppm and 20ppm showed the samples were clear and bright. It can conclude that the scale inhibitor gave very good results at 10ppm and 20ppm.

Table 14 shows the results obtained from Well No. Q-25 in the table above it is observed that the 20 ppm dose rate of scale inhibitor gave a very good result at 95^{o}C.

Table 15: Scale inhibitor (DTPMP) Static jar test summary – well No. Q-25

Dose Rate (ppm)	**40^{o}C**	**60^{o}C**	**80^{o}C**	**95^{o}C**
0.00	0.00	0.00	0.00	0.00
3	96.23	83.46	75.50	60.00
5	97.26	87.92	81.26	71.50
7	98.90	97.67	88.00	77.50
10	100.00	98.20	94.83	88.50
20	100.00	99.20	98.80	93.30

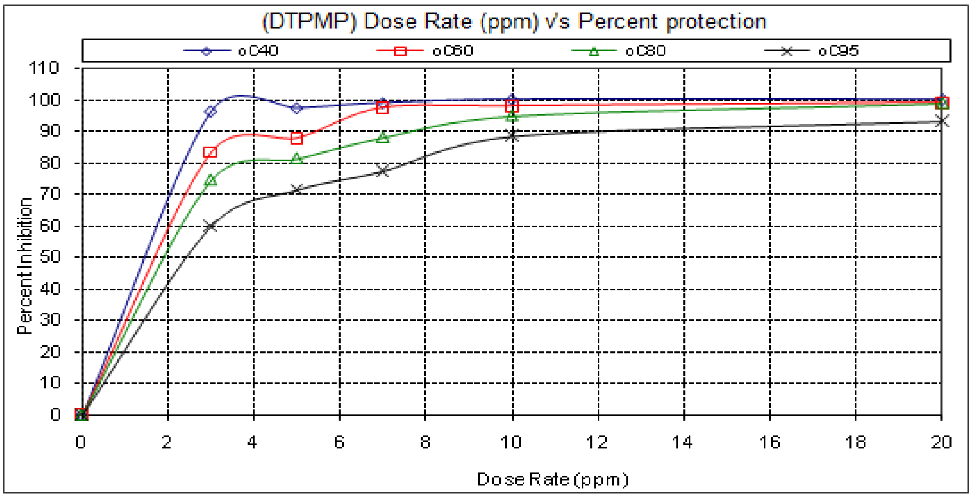

Figure 6: shows the relation between concentrations dose and temperatures

Table 15 and figure 6 show that the dosages rate versus the percent of the Scale inhibition (DTPMP) in various temperature (40, 60, 80 and 95°C), and from these data it conclude that the 7ppm and 10ppm are the good inhibition and economically cost as well the best inhibition at all temperatures was clear at concentration of 20 ppm.

3.3. Dynamic Scale Rig Test Results:

3.3.1. The efficiency of scale inhibitor at different concentrations for Well No. Q-25

The Dynamic test has also done for the sample by using dynamic scale rig test at a temperature of 75°C and 2 liter per hour injection rate of sample (scaling solution) to determine the efficiency of scale inhibitor (DTPMP) at injection rates of (3, 5, 7, 10, and 20ppm) respectively as shown in figures (7(a),7(b)). And through this test, the scale inhibitor (DTPMP) achieved a very good efficiency at injection rates from 7ppm to 10ppm.

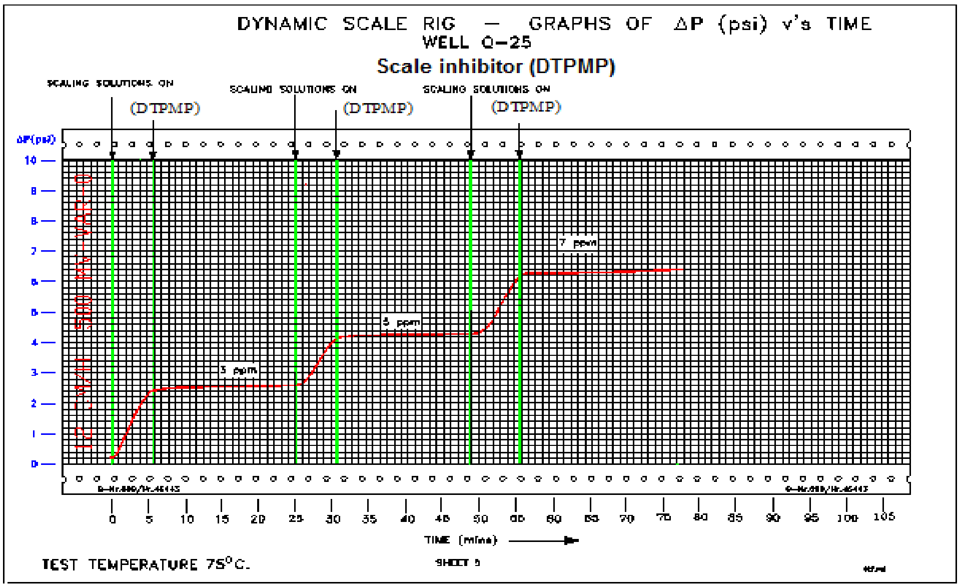

Figure 7 (a): dynamic test of water from well No. Q-25 at different concentration of (DTPMP) at 3, 5 and 7 ppm

It has been noticed that the rate of change in pressure with respect to the time is constant, and this proves the scale inhibitor (DTPMP) achieved a very good efficiency as it managed to prevent formation of the calcium carbonate $CaCO_3$ scale through a time period with respect to the injection of both the scale inhibitor and sample (scaling solution).

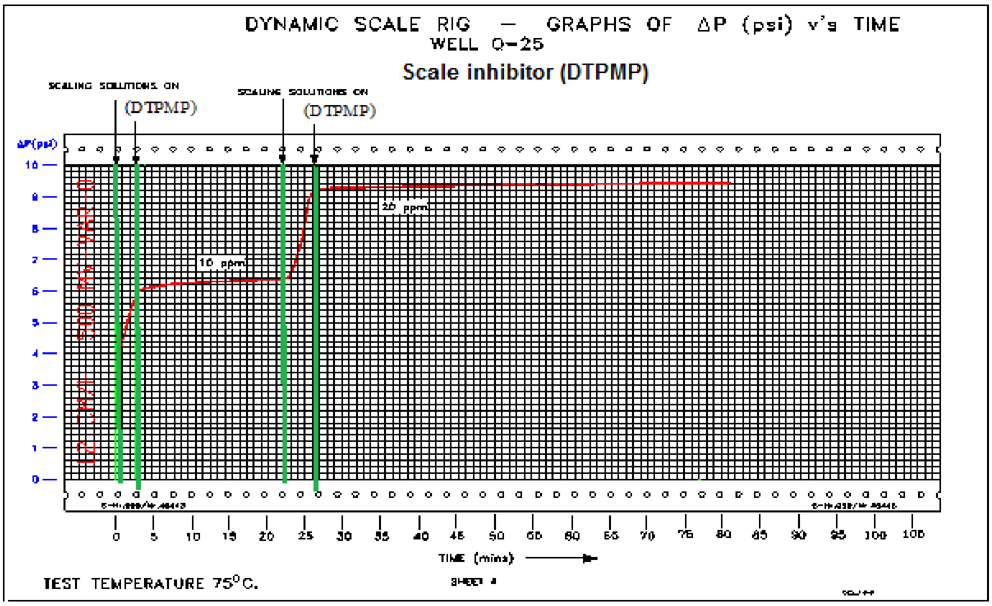

Figure 7 (b): dynamic test of water from well No. Q-25 at different concentration of (DTPMP) at 10 and 20 ppm.

IV. Conclusion and Recommendation

Throughout the results illustrated in the laboratory of both static jar and dynamic test, which have been done for sample of well (Q-25) Waha oil field, and to identify the efficiency of the scale inhibitor (DTPMP) in eliminating the scale of calcium carbonate $CaCO_3$ we found the following :Sample of well Q-25 has been done to determine the efficiency of scale inhibitor (DTPMP), at different temperatures (40, 60, 80 and 95°C) and scale inhibitor injection rates (3, 5, 7, 10, and 20 ppm). The experiments shows that the scale inhibitor (DTPMP) is effective in preventing formation of calcium carbonate $CaCO_3$ scale, where it has achieved efficiency from 94.83% to 98.20% at concentration of scale inhibitor of 10ppm. The Dynamic test has cared out at 75°C and 2 liter per hour injection rate of scaling solution (well Q-25) to determine the efficiency of scale inhibitor (DTPMP) at concentration (3, 5, 7, 10 and 20 ppm) as shown in figures (7 (a) and 7 (b)). It has been noticed that the rate of change in pressure with respect to the time is constant, and this proves that the scale inhibitor (DTPMP) achieved a very good efficiency as it managed to prevent formation of calcium carbonate $CaCO_3$ scale through a time period with respect to the injection of both the scale inhibitor and sample (scaling solution). The scale inhibitor (DTPMP) achieved a very good efficiency at injection rates of 10ppm.

We conclude that the scale inhibitor (DTPMP) is very efficient in preventing calcium carbonate scale. It is advisable to inject at concentration rate of 10 ppm to provide economic wais and better performance.

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