An In-depth Study of Calcium Carbonate Scale Formation and Inhibition

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Abstract

A fundamental study of scale formation of calcium carbonate ($CaCO_3$) for producing oil wells has been carried out. This article presents the study of the prediction of salt deposition in two different synthetic formation waters and investigates the effects of temperature and pressure on calcium carbonate precipitation. The dependence of the induction period of the precipitation of calcium carbonate on the concentration of calcium ions was studied. In order to study the chemical scale inhibition, the most effective inhibitors, which are based on the risk analysis of scaling and laboratory reagent selection, were examined for evaluating the performance of salt inhibition. In this work, a new multi-component inhibitor was made and its scale effectiveness was evaluated at different concentrations. The developed inhibitor was mixed with other inhibitors to prevent calcium carbonate precipitation. The observations showed the synergetic inhibition effect on the scale inhibition at different concentrations of scale inhibitors. The new inhibitor provided high scale effectiveness at specific concentrations and low corrosion activity.

Keywords: Calcium Carbonate, Corrosiveness, Induction Period, Scale Inhibition, Synergetic Effect

1. Introduction

A supersaturated calcium carbonate aqueous solution can be formed resulting from incompatible mixing of injected water (on the basis of calcium chloride) with formation water (hydrocarbonate-sodium type) (Nasr-El-Din et al., 2006). The precipitation of the salt occurs by increasing the amount of calcium ions in the mixture and decreasing the amount of dissolved CO_2 in the formation water. This leads to the precipitation of excess amounts of carbonate in the wellbore and production facilities.

The violation of the established equilibrium of water occurs due to pressure reduction during production (Ramstad et al., 2004; Kan and Tomson, 2010), and then a new relation of dissolved CO_2 is established between water and oil phases. The reduction of dissolved CO_2 in the water results in the precipitation of calcium carbonate from the saturated media by salt-forming ions. The process is intensified when the pressure is below the saturation pressure.

Calcium carbonate solubility is significantly reduced with increasing temperature (Weyl, 1959). Therefore, the possibility of $CaCO_3$ deposition increases with increasing temperature. The effect of the temperature can be explained by the formation of carbonate precipitate in some deep and high

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temperature wells, where the injected water is saturated with calcium carbonate under reservoir conditions.

The solubility of the calcium carbonate is significantly affected by the pH of the media (Economou et al., 1996). In acidic conditions the calcite solubility is significantly higher than alkaline conditions (Kelland, 2009). The possibility of the carbonate precipitation increases with increasing pH. This is due to the fact that the solubility of the dissolved CO_2 is also dependent on the pH of aqueous solution.

To effectively combat the deposition of salts, a systematic approach is required. An overall strategy to combat scaling potentials should be considered at two levels. The first level is a program, including determining the possible accumulation of deposited salts in the wells and the determination of the saturation index (SI) (Oddo and Tomson, 1994). This is necessary in order to predict the level of scaling in the wells and to evaluate the risks and losses in the application of inhibitors of salt depositions. The second level offers an extended analysis of scaling risks. It includes the modeling of injection water and rock interaction. The model tracks the changes in the ionic composition of water injected to producing wells when it is mixed with formation water in motion (Sorbie and Mackay, 2000). The second level is designed for long-term forecasting of salt capacity through geological and hydrodynamic models of the field.

Scaling leads to the reduction in the productivity of wells, premature failure of the downhole equipment, unscheduled workover, and, as a consequence, a significant deterioration of the technical and economic indicators of oil and gas companies (Crowe et al., 1994, Khormali and Petrakov, 2014). Currently, the reagent protection of oilfield equipment from deposits of inorganic salts is the most common and effective method of combating scaling (Cheremisov et al., 2008; Yakimov et al., 2010). Technologically, a complete prevention of the deposition of inorganic salts including calcium carbonate can be achieved with the proper choice of inhibitors and their application to the entire path of the well through an appropriate technology.

Scale inhibitors can be divided into the following three types depending on their mechanism of action: 1) Chelation, a substance that binds the salt-forming cations and prevents their interaction with the salt-forming anions (Vetter, 1972). 2) Threshold inhibitors, the addition of which to the solution prevents the initiation and growth of salt crystals (Cooper et al., 1979). 3) The third type of scale inhibitors is a crystallographic destroying inhibitor, which does not prevent the crystallization of salts, but only modifies the shape of the crystals.

The mechanism of scale inhibitors is based on the adsorption processes. Inhibitors suppress the growth of the crystal, alter its shape and dimensions, and degrade the adhesion of the crystal when adsorbing on the centers of the salt compound (Tomson et al., 2003). Complex making reagents have been effectively used to prevent the crystallization of poorly soluble inorganic salts.

Amongst carbonate deposit inhibitors, the phosphonate types are found to be the most effective (Voloshin et al., 2003). Calcium carbonate scale inhibition was studied through using three different inhibitors polyacrylic acid, aminotrimethylenephosphonic acid, and polyamino polyether methylenephosphonate (Tang et al., 2008). The effects of temperature and concentration of polyphosphates, polyphosphonate and polycarboxylate inhibitors on calcium carbonate precipitation were evaluated and only polyphosphonates remained efficient at high temperatures (Ketrane et al., 2009). In this paper, an inhibitor, which is based on the polyethylene polyamine-N-methylphosphonic and oxiethilidendiphosphone acids, is studied at high temperatures.

The objective of this study is the investigation of calcium carbonate precipitation in formation waters depending on the pressure and temperature and its scale inhibition by experiments. The formation of calcium carbonate is considered by the induction period measurement of ions. In this work, different inhibitors are examined for the optimum prevention of calcium carbonate precipitation with the lowest corrosion activity.

2. Materials and methods

Two different synthetic waters, which contained various ion concentrations, were used in this study. Working solutions were prepared to investigate the spontaneous deposition of calcium carbonate in aqueous solutions. Solutions of calcium chloride and sulfuric acid at various concentrations were used in the experiments. Throughout the experiments, the solutions were constantly mixed with a magnetic stirrer. The experiments were performed on a laser analyzer of microparticles. Light intensity data, which transmit through the cuvette with a supersaturated solution, were obtained in the result of the laser device. The beginning of precipitation appeared by changing the initial constant light intensity. The induction period of the precipitation was determined by changing the slope of the recorded absorbance curve depending on the time (He et al., 1999; Waly et al., 2012). The induction period of precipitation (τ) depends on the initial concentration of calcium ions, which in turn is determined by the supersaturation of the solution. The induction period can be calculated from Christiansen and Nielsen equation (Equation 1) (Christiansen and Nielsen, 1951; Christiansen, 1954; Olsson, 1995):

$$\tau = k[Ca^{2+}]_0^{1-n} \tag{1}$$

The driving force of the deposition (β) can be explained as the difference of the chemical potential between crystallization substances in the crystal and in a supersaturated solution (Vincent et al., 1984; Wiechers et al., 1975):

$$\beta = \frac{-\Delta\mu}{R(\frac{T-32}{1.8}+273)} \tag{2}$$

To predict the calcium carbonate formation in the absence of gas phase, the pH value of water and the chemical (ion concentration and ionic strength of the solution) and thermodynamic properties of formation water (pressure and temperature) are used. The saturation index of calcium carbonate in the absence of gas phase is defined as follows (Moghadasi et al., 2003):

$$pH = \log[\frac{[HCO_3^-]}{Ca_{aq}^{CO_2}}] + 6.39 - 1.198 \times 10^{-3}T + 7.94 \times 10^{-6}T^2 - 3.53 \times 10^{-5}P - 1.067S_i^{0.5} + 0.599\,S_i \tag{3}$$

$$SI = \log[\frac{[Ca^{2+}][HCO_3^-]}{Ca_{aq}^{CO_2}}] + 3.63 + 8.68 \times 10^{-3}T + 8.55 \times 10^{-6}T^2 - 6.56 \times 10^{-5}P - 3.42S^{0.5} + 1.373\,S_i \tag{4}$$

The efficacy of scale inhibition is calculated by Equation 5:

$$E(\%) = \frac{m_0 - m}{m_0} \times 100 \tag{5}$$

The establishment of multi-component mixtures of various scale inhibitors with synergetic inhibition effect is a more economical and highly efficient way of expanding the range of scale inhibitors and improving of their quality. There are certain principles of combining inhibitors to produce composite inhibitors with a synergetic effect, which have an efficiency value greater than the sum of the effects

of the initial components. These composite inhibitors are prepared from empirical selection. However, composite inhibitors of different chemical nature (Liu et al., 2012) should be used in the synergistic multicomponent systems.

Scale inhibitors should have low corrosion activity. The corrosiveness of the developed composite scale inhibitors is evaluated through the mass reduction of metal samples after their immersion in the inhibitor solution. The corrosion rate is given by:

$$V = 8760 \frac{m_1 - m_2}{St\rho} \tag{6}$$

3. Results and discussion

The scale inhibitor studies were conducted in laboratory conditions and the formation of calcium carbonate was modeled and monitored. The spontaneous formation of solid particles in a solution from the standpoint of thermodynamics may occur only when the overall energy of the system decreases. Phase formation, including the formation of precipitates in solutions, occurs only in a metastable system, a condition, which often differs substantially from equilibrium (Kan and Tomson, 2010).

The stoichiometry of the salt components, the molar ratio of calcium and carbonate ions, does not affect the induction period of precipitation. The experimental results, which were obtained from the spontaneous precipitation of calcium carbonate in an aqueous solution, are shown in Figures 1 and 2. As shown in Figure 1, the induction period curves practically coincide for different ratios of the components (50:1 and 1:50), i.e. the system is invariant.

The stability of calcium carbonate solution is highly dependent on the driving force. Figure 1 shows that spontaneous deposition practically does not occur in the supersaturated solutions for a long time when $\beta < 5$.
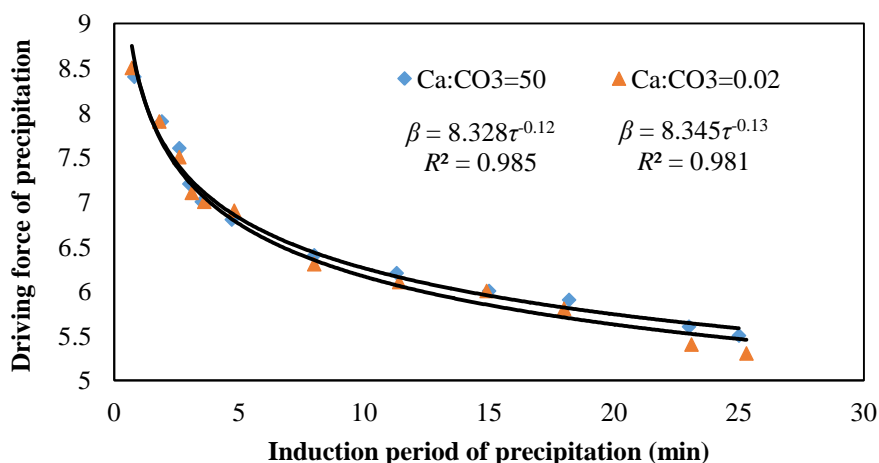

Figure 1
The dependence of driving force of scale precipitation on the induction period of precipitation.

As shown in Figure 2 and according to Equation 1, the number of calcium ions (n) participating in the formation of the critical nucleus of a salt crystal is equal to three. The study of the crystal nucleation and particle growth of crystals allows a deeper understanding of the kinetics of formation of scaling in spontaneous precipitation in supersaturated solutions. These results are important and can lead to the

ability to control the formation of sparingly soluble salts, such as calcium carbonate, in the operation of oil wells.
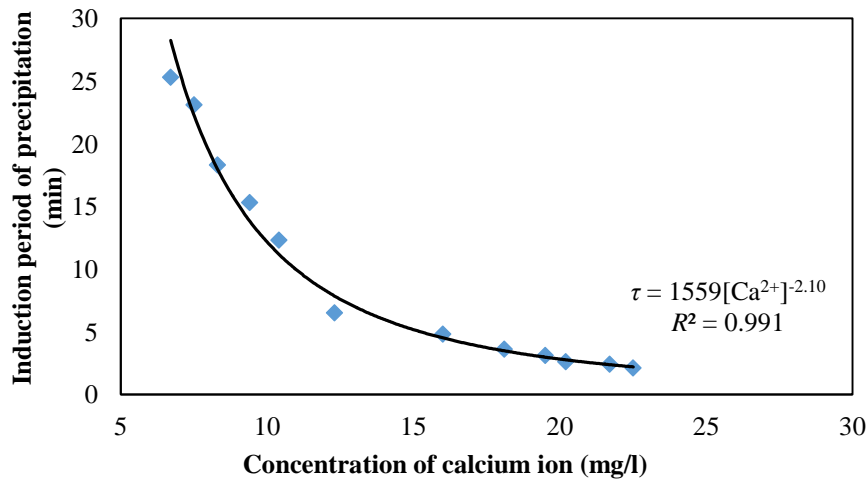

Figure 2
The dependence of the induction period of precipitation on the initial concentration of calcium.

The used synthetic formation waters are predominantly of chloride-calcium type. In this regard, the ion amount of hydrocarbonate and calcium in the water exerts a decisive influence on the carbonate precipitation (Ostroff, 1967). Knowing the quantitative relationship between the amount of these ions and the dissolved CO_2 present in the formation water, in compliance with the equilibrium ratio, makes it possible to predict the appearance of carbonate precipitation (Kan and Tomson, 2010). The results of water analysis are presented in Table 1. As shown in this table, the bicarbonate ions and calcium ions, which form the calcium carbonate precipitation, were found in both of the waters.

Table 1
Analysis of synthetic formation waters at a temperature of 75 °C.

Parameters		First water	Second water
	HCO_3^-	1764	2781
	CO_3^{2-}	592	715
	Cl^-	43947	51758
	SO_4^{2-}	217	183
	Li^+	0	1
Ion content (mg/l)	Ca^{2+}	11272	8965
	Mg^{2+}	486	739
	Na^+	12854	10493
	Sr^{2+}	0	12
	Ba^{2+}	73	9
Total dissolved salts (g/l)		71.205	75.656
pH		6.9	7.6
Density (kg.m^{-3})		1048	1061

Salt precipitation cannot be considered as the result of a simple isothermal mixing of formation and injected water in the oilfield development during water flooding. To increase the effectiveness of the prevention of salt deposition timely and reliable prediction of salt formation is required (Rocha et al., 2001). In this study, the composition of the injected and formation waters was analyzed. The results of scaling prediction are shown in Figure 3. Each diagram comprises of two zones; the active zone for scale formation is located above the curve in both of the waters in the diagrams.

Figure 3 illustrates a higher pressure for the salt deposition of the first synthetic formation water in comparison with the second water. As shown in Figure 3, for temperatures higher than 20 °C, salt deposits at lower pressures. It means that with pressure drop, the possibility of salt precipitation increases in a wide range of temperatures. At high pressures, scale forms just at high temperatures. Figure 3 is shown as a good sample for production by electric submersible pumps (ESP's) because increasing temperature in these pumps is caused by the working motor as well as the reduction of pressure in the formation during production.
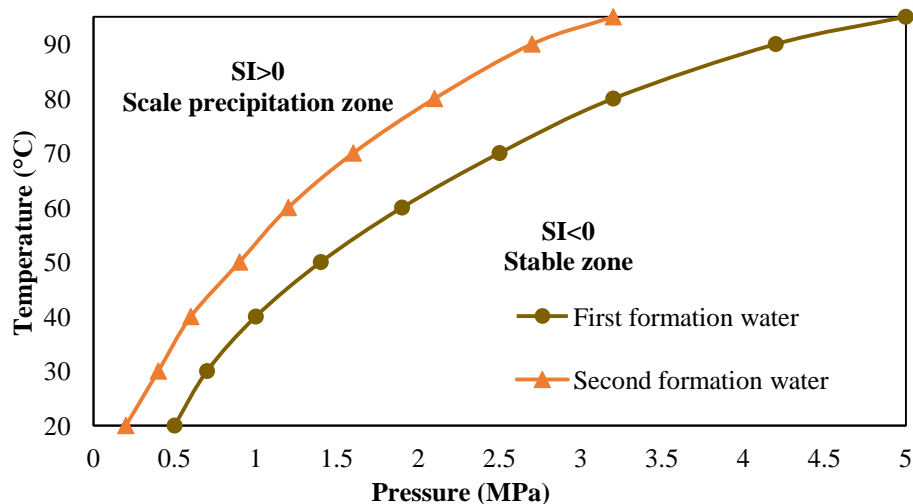

Figure 3
Pressure-temperature phase diagram of calcium in formation waters.

The effectiveness of scale inhibition largely depends on the composition and structure of the salts (Khormali and Petrakov, 2014). The selection of an effective inhibitor for scale inhibition is the most important factor in the development of technologies of inhibition. The developed or selected inhibitor is applied to specific parameters of the operation well and the chemical composition of the producing water. The used inhibitors for oil well equipment protection are presented in Table 2. Inhibitor No. 1 is the newly developed inhibitor, which is made for the prevention of calcium carbonate scale in the equipment, especially in ESP's.

Table 2
Composition of the scale inhibitors of calcium carbonate.

Inhibitor number	Composition
1 (new inhibitor)	Mixture of $C_2H_8O_7P_2$ (3%), NH_4Cl (4%), $C_{12}H_5N_7O_{12}$ -N- CH_5O_3P (4%), HCl (10%), C_3H_8O (2%) and water – remainder
2	Mixture of $H_5P_3O_{10}$, C_2H_7NO, $(CH_3)PO(OH)_2$ and $C_{16}H_{32}O_6$
3	Mixture of $(CH_3)PO(OH)_2$, $K_4P_2O_7$, C_2H_7NO and CH_6NO_3P

As shown in Table 2, this inhibitor is an aqueous solution made of three acids, ammonium chloride, and isopropyl alcohol. These components are mixed in mass percentage. The inhibitor No. 1 is a liquid with a density of 1095 kg.m^{-3} at 20 ℃. The pH value of this inhibitor is equal to 7.3 and its kinematic viscosity is 36 cSt at 20 ℃; the inhibitor No. 1 has a freezing point of -40 ℃ and is readily soluble in water. This inhibitor has a good compatibility with the produced waters, which contain high salinity. Scale inhibitors No. 2 and No. 3 are industrial inhibitors, which are injected into the oil wells for the protection of calcium carbonate precipitation. Inhibitor No. 2 contains esters, amino alcohols, and nonionic surfactant. The third inhibitor is made of two different kinds of acids, potassium pyrophosphate, and amino alcohols. The highest scale inhibition of calcium carbonate occurred at ratios that are presented for the inhibitor No. 1 in Table 2.

The efficiency of scale inhibitors was obtained as the percentage of total amounts of calcium that is kept in the solution by the inhibitor. The solutions of formation waters with scaling inhibitors were heated for 5 hours at a temperature of 75 ℃. After thermostating and cooling, the precipitate was filtered off and the amount of calcium ion was determined. Scale inhibitor effectiveness was calculated from Equation 5 and is shown in Figures 4 and 5 for formation waters. The new inhibitor (No. 1) has the highest effectiveness in both synthetic formation waters. The scale inhibition effectiveness for this inhibitor reaches a value of 90%. Figures 4 and 5 show that, at concentrations higher than 30 mg/l, the growth of scale inhibitor effectiveness is not significant for the inhibitor No. 1. Figure 5 shows that the inhibitor No. 3 protects better than the inhibitor No. 2 at low concentrations for the second formation water.
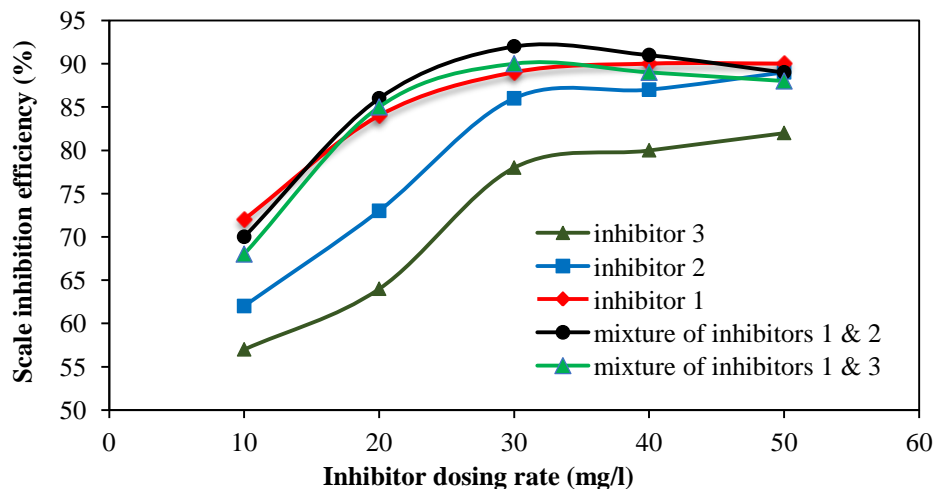

Figure 4
Efficiency of scale inhibitors in first synthetic formation water.

The inhibitor No. 1 is used as a reference inhibitor for synergetic inhibition effect consideration because of its higher scale inhibitor effectiveness in comparison with the two other inhibitors in both formation waters. For the practical use of mixtures of scale inhibitors, it is necessary to select compounds with the highest synergetic inhibition effect. Both mixtures of inhibitors were prepared in a mass-mixing ratio of 60:40. Figure 4 shows that the mixture of inhibitor No. 1 and inhibitor No. 2 gives the highest efficiency for the first formation water at the concentration of 30 mg/l. This means that the mixture has a synergetic inhibition effect at this concentration. This mixture has efficiency less than that of inhibitor No. 1 in the range of concentration of 10-15 and 45-50 mg/l. As shown in Figure 5, for the second formation water, the mixture of inhibitors No. 1 and No. 2 has scale

inhibition effectiveness greater than that of the inhibitor No. 1 at concentrations equal to or greater than 15 mg/l. The highest value occurred at the concentration of 30 mg/l. In this water, the mixture of inhibitors No. 1 and No. 3 is more effective in comparison with the inhibitor No. 1 in a small range of concentrations; but the difference is not high in this range. Therefore, according to the Figures 4 and 5, the most effective and economic inhibitor is the mixture of inhibitors No. 1 and No. 2 at the concentration of 30 mg/l in a mass ratio of 60:40 respectively.
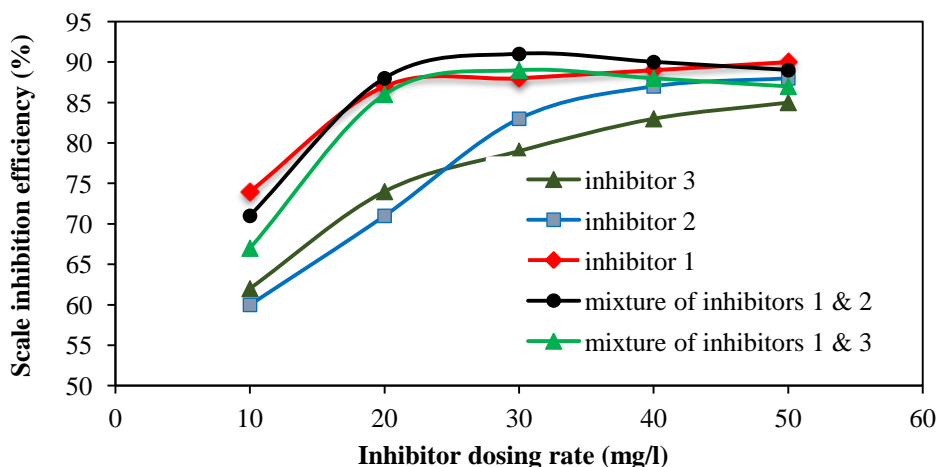

Figure 5
Efficiency of scale inhibitors in second synthetic formation water.

The corrosivity test of the reagents was carried out by the gravimetric method using steel plates with dimensions $4 \times 1.2 \times 0.2$ cm, by aging for 72 hours at 20 ºC. The results of the corrosivity determination of the investigated compounds from Equation 6 are given in Table 3. The used metal sample has a density (ρ) of 7821 kg.m^{-3}.

The rate of corrosion of the equipment in contact with the scale inhibitor in the well must not exceed 0.1 mm/yr (Khormali and Petrakov, 2014). As shown in Table 3, the inhibitor No. 1 has the lowest corrosion rate in both of the formation waters, whereas the inhibitor No. 3 gives rise to the highest corrosion activity among the considered inhibitors. The mixture of inhibitors No. 1 and No. 2 has a corrosion rate higher than that of the first inhibitor but lower than that of the second inhibitor. From the data in Table 3, it can be noted that all of the above chemical composite inhibitors showed an allowable corrosion rate and could be considered as inhibitors to prevent scaling of calcium carbonate in the wells.

Table 3
Corrosion activity of the scale inhibitors.

Scale inhibitor	First water			Second water		
	m_1 (g)	m_2 (g)	V (mm/yr)	m_1 (g)	m_2 (g)	V (mm/yr)
1	7.4736	7.4723	0.042	7.4618	7.4603	0.047
2	7.4736	7.4715	0.069	7.4618	7.4598	0.065
3	7.4736	7.4713	0.074	7.4618	7.4596	0.072
Mixture 1 & 2	7.4736	7.4720	0.049	7.4618	7.4601	0.055
Mixture 1 & 3	7.4736	7.4717	0.061	7.4618	7.4599	0.063

4. Conclusions

The features of the crystallization kinetics of calcium carbonate were researched from supersaturated aqueous solutions in this study. It established that the duration of the induction period is well approximated (R^2=0.991) by a power dependence of the initial concentration of calcium ions in the solution, and it is practically independent of the stoichiometry of the salt components. The most effective scale inhibitor for the prevention of calcium carbonate deposition is the mixture of inhibitor No. 1 and inhibitor No. 2 at the concentration of 30 mg/l. This mixture was used at a mass ratio of inhibitor No. 1 to inhibitor No. 2 equal to 60:40, and at this mass ratio a synergetic inhibition effect was observed. The developed mixture of inhibitors can be injected to prevent calcium carbonate precipitation into oil wells. ESP is used because the results of pressure-temperature diagrams show that, at low pressure and in a wide range of temperatures (higher than 20 ºC), calcium carbonate is precipitated. Applying the prepared inhibitor in oil wells can increase ESP run life. There was no pitting on the surface of metal samples and their corrosion rate value was less than 0.1 mm/yr in contact with the scale inhibitors in the formation waters. Therefore, the developed composite inhibitors have permitted corrosion activity and can be used as reagents to prevent calcium carbonate precipitation in oil wells. The results of the experiments can be used for the effective protection of equipment with minimum corrosion activity in oil wells with calcium carbonate precipitation. The presence of paraffin and wax in oil wells affects the values of saturation index and the induction period of precipitation; therefore, the consideration of their effects is suggested for further works. The effects of other scale inhibitors on the effectiveness of the prepared scale inhibitor are not considered in this paper. Conducting experiments on oil wells, where there are several types of salts including calcium carbonate, is therefore recommended.

Nomenclature

E	: Efficiency of scale inhibition [%]
k	: Proportionality factor [--]
m	: Mass of salt precipitate in the solution with inhibitor [g]
m_0	: Mass of salt precipitate in the solution without inhibitor [g]
m_1	: Mass of the metal sample before corrosion test [g]
m_2	: Mass of the metal sample after corrosion test [g]
n	: Number of ions in the critical nucleus [--]
P	: Pressure, [psi]
R	: Gas constant [8.314 $J.K^{-1}.mol^{-1}$]
S	: Surface area of metal sample [m^2]
S_i	: Ionic strength, [M]
SI	: Saturation index [--]
t	: Time of corrosion inhibition test [hr]
T	: Temperature [°F]
V	: Corrosion rate [$mm.yr^{-1}$]
Greek symbols	
β	: Driving force [--]
$\Delta\mu$	: Difference in chemical potential [V]
ρ	: Density of metal sample [$kg.m^{-3}$]
τ	: Induction period time [min]

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