HSR (LONDON),
HOUGHTON STREET REVIEW JOURNAL
ISSN: 2058-5519|Impact factor-15.9
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SALT DEPOSIT DISSOLUTION TECHNOLOGY: SCIENTIFIC BASIS FOR SELECTING CHELATING AND CONVERSION FORMULATIONS

Eshonqulov Nurbek Qaxramon o‘g‘li
Tashkent Institute of Chemical Technology, Uzbekistan
E-mail: nurik1995.nurbek@gmail.com
ORCID: https://orcid.org/0009-0009-3142-7852
Absalyamova Gulnoza Mamatkulovna
Associate Professor, Department of Chemical Technology of Oil and Gas Processing, Tashkent Institute of Chemical Technology, Uzbekistan
E-mail: g.absalyamova@tkti.uz; absalyamovagulnoza@gmail.com
ORCID: https://orcid.org/0000-0003-0275-0414
doi:https://doi.org/10.5281/zenodo.23018216

Abstract. The article analyses the technology of chemical dissolution of carbonate and sulfate salt deposits by comparing published experimental data, recalculating the composition of the Chegara field formation water into equivalent form and performing a stoichiometric assessment. It is shown that the Chegara water is of the calcium chloride type (rNa/rCl = 0.85) with a total divalent cation content of 170 mmol/L, so that a 0.5 M chelating dissolver prepared with this water loses ≈34 % of its capacity. It is substantiated that the K_4-EDTA + K_2CO_3 system (pH 10.5) is more effective for calcium sulfate and catalysed DTPA systems for barium sulfate, while biodegradable MGDA and GLDA outperform DTPA and HEDP in gypsum dissolution. A six-stage process scheme is proposed.

Keywords: salt deposits, gypsum, barite, calcite, scale dissolver, EDTA, DTPA, MGDA, conversion, formation water.

ТЕХНОЛОГИЯ РАСТВОРЕНИЯ СОЛЕВЫХ ОТЛОЖЕНИЙ: НАУЧНЫЕ ОСНОВЫ ВЫБОРА КОМПЛЕКСООБРАЗУЮЩИХ И КОНВЕРСИОННЫХ СОСТАВОВ

Эшонкулов Нурбек Кахрамон угли,
Абсалямова Гульноза Маматкуловна,
Ташкентский химико-технологический институт, Узбекистан

Аннотация. Технология химического растворения карбонатных и сульфатных солевых отложений проанализирована на основе сравнения экспериментальных данных, пересчёта состава пластовой воды месторождения Чегара в эквивалентную форму и стехиометрической оценки. Показано, что вода месторождения Чегара хлоркальциевого типа (rNa/rCl = 0,85), сумма двухвалентных катионов – 170 ммоль/л, и при приготовлении на ней 0,5 М хелатного

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растворителя теряется ≈34 % его ёмкости. Обосновано, что для сульфата кальция эффективнее система K_4-ЭДТА + K_2CO_3 (pH 10,5), для сульфата бария – ДТПА с катализаторами, а биоразлагаемые MGDA и GLDA превосходят ДТПА и ОЭДФ при растворении гипса. Предложена шестиэтапная технологическая схема.

Ключевые слова: солевые отложения, гипс, барит, кальцит, растворитель отложений, ЭДТА, ДТПА, MGDA, конверсия, пластовая вода.

TUZLI CHO‘KINDILARNI ERITUVCHI TEXNOLOGIYASI: KOMPLEKS HOSIL QILUVCHI VA KONVERSION TARKIBLARNI TANLASHNING ILMIY ASOSLARI

Eshonqulov Nurbek Qaxramon o‘g‘li,
Absalyamova Gulnoza Mamatkulovna,
Toshkent kimyo-texnologiya instituti, O‘zbekiston

Annotatsiya. Maqolada karbonat va sulfat tarkibli tuzli cho‘kindilarni kimyoviy eritish texnologiyasi eksperimental ma’lumotlarni qiyoslash, Chegara koni qatlam suvi tarkibini ekvivalent shaklga qayta hisoblash va stexiometrik baholash asosida tahlil qilingan. Chegara koni suvi xlorkalsiyli (rNa/rCl = 0,85), ikki valentli kationlar yig‘indisi 170 mmol/l ekani, bu suvda tayyorlangan 0,5 M xelat eritgich sig‘imining ≈34 % i yo‘qotilishi ko‘rsatilgan. Kalsiy sulfat uchun K_4-EDTA + K_2CO_3 (pH 10,5), bariy sulfat uchun katalizatorli DTPA tizimlari samaraliroq, biologik parchalanuvchan MGDA va GLDA esa gipsni eritishda DTPA va HEDP dan ustun ekani asoslangan. Olti bosqichli texnologik sxema taklif etilgan.

Kalit so‘zlar: tuzli cho‘kindi, gips, barit, kalsit, cho‘kindi eritgich, EDTA, DTPA, MGDA, konversiya, qatlam suvi.

INTRODUCTION

Salt deposits are solid layers formed by the crystallisation of sparingly soluble salts from formation and process waters (calcite $CaCO_3$, gypsum $CaSO_4 \cdot 2H_2O$, anhydrite $CaSO_4$, barite $BaSO_4$, celestine $SrSO_4$) on metal and rock surfaces. By narrowing the flow area of pipelines, pumps and perforations, they cause premature equipment failure and costly workovers [1; 2]. In heat-exchange equipment the same process takes place as scaling and reduces heat transfer [3].

The problem is particularly relevant in Uzbekistan. In the formation water sampled at the SP-1 gathering point of the Chegara field in Western Uzbekistan, the dry residue is 149,358 mg/L, calcium 5410.8 mg/L and total hardness 340 meq/L [4]. The annual demand of the Navoi Mining and Metallurgical Combine for scale inhibitors is 140 thousand tonnes and is met mainly by the imported reagent Komperex-710 [5]. Presidential Resolution No. PP-4265 of 3 April 2019 approved the programme for the development of the chemical industry for 2019–2030, and Resolution No. PP-4992

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of 13 February 2021 defined the production of high value-added chemical products as a priority task [6; 7].

Scale control is carried out in two directions: preventing scale formation (inhibitors, magnetic and acoustic treatment, coatings) and removing deposits that have already formed [2; 4]. An inhibitor does not remove an accumulated layer – this task is performed by dissolvers. Carbonates readily dissolve in hydrochloric, acetic, formic and sulfamic acids, whereas sulfates are acid-resistant: at 25 °C the solubility of calcium sulfate in hydrochloric acid does not exceed 1.8 wt %, and it reprecipitates once the acid is spent [8; 9]. Barium sulfate is practically insoluble in hydrochloric, formic, citric and acetic acids [10].

For this reason, dissolvers based on aminopolycarboxylic acids (EDTA, DTPA, HEDTA), biodegradable GLDA and MGDA, and carbonate converters are being actively studied [11; 12; 13]. However, the results were obtained under different conditions, and the task of linking them to the composition of local field waters and turning them into practical selection criteria remains open.

The aim of the study is to carry out a comparative analysis of experimental data on salt deposit dissolution technologies, to substantiate dissolver selection criteria taking into account the composition of local formation water, and to develop a process scheme.

MATERIALS AND METHODS

A systematic analytical approach was used. Sources were selected according to three criteria: publication in a peer-reviewed journal, SPE conference proceedings or a patent; quantitative reporting of dissolution efficiency or capacity; and clear specification of temperature, time, pH and reagent concentration. Seven key experimental sources (1991–2025) meeting these criteria were included in the comparative table. The ionic composition of the formation water at the SP-1 gathering point of the Chegara field [4] was taken as the local object, and the content of each ion was converted into milligram-equivalents:

$$e_i = \frac{C_i \cdot z_i}{M_i} \quad (1)$$

where C_i is the ion concentration, mg/L; z_i is the ion charge; M_i is the molar mass, g/mol. The sum of sodium and potassium was found from the difference between anion and cation equivalents. The water type was determined according to V.A. Sulin's classification from the ratios rNa/rCl and (rCl – rNa)/rMg. The theoretical specific consumption of the dissolver (kg of reagent per 1 kg of scale) was calculated from the reaction stoichiometry:

$$G = \frac{\nu \cdot M_r}{M_s \cdot w} \quad (2)$$

where ν is the number of moles of reagent per 1 mol of scale; M_r and M_s are the molar masses of the reagent and the scale; w is the mass fraction of the reagent (w = 1 for a pure substance). In the sources, dissolution efficiency was determined gravimetrically from the initial (m_0) and undissolved (m_1) masses of the scale:

$$E = \frac{m_0 - m_1}{m_0} \cdot 100\ \% \quad (3)$$

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Temperatures on the Fahrenheit scale were converted to the Celsius scale, and corrosion rates from lb/(ft^2·year) to kg/(m^2·year) (1 lb/ft^2 = 4.882 kg/m^2).

RESULTS

Scale types and their response to dissolvers. The main scale types and the reagents acting on them are summarised in Table 1.

Table 1

Main salt deposits and agents for their dissolution

Scale	Formula	Solubility and properties	Effective dissolvers
Calcite	$CaCO_3$	Readily soluble in acids with CO_2 release	HCl (10–15 %), acetic, formic and sulfamic acids; EDTA, GLDA [8; 11]
Gypsum	$CaSO_4 \cdot 2H_2O$	In water 2.36 kg/m^3 (25 °C), 1.69 kg/m^3 (90 °C); in HCl ≤ 1.8 %	EDTA (up to 43 g/L), K_4-EDTA + K_2CO_3, MGDA, GLDA [8; 3; 12]
Anhydrite	$CaSO_4$	Stable above 98 °C; dissolves in EDTA up to 34 g/L	K_4-EDTA + K_2CO_3, DTPA [8; 3]
Barite	$BaSO_4$	Practically insoluble in water and acids	K_5-DTPA + K_2CO_3 / KCl / HCOOK [10]
Celestine	$SrSO_4$	Insoluble in acids	DTPA, HEDTA [13]
Iron sulfide	FeS	Soluble in acid (H_2S is released)	HCl + iron-sequestering agent; 40 % K_5-DTPA – 30 kg/m^3 [14]

The choice of dissolver depends primarily on the anionic composition of the scale: carbonate and iron sulfide dissolve in acid, whereas sulfates dissolve only in chelating or conversion systems. Since gypsum solubility decreases with increasing temperature, gypsum accumulates intensively in the hot zone of the well and on heated surfaces [8].

Analysis of the Chegara field formation water. The results of recalculating the primary data by formula (1) are given in Table 2.

Table 2

Ionic composition of the Chegara field (SP-1) formation water

Parameter	mg/L	meq/L	Equivalent fraction, %	mmol/L
Chloride, Cl^-	76 913.0	2169.4	49.51	2169.4
Sulfate, SO_4^{2-}	397.0	8.27	0.19	4.13
Bicarbonate, HCO_3^-	817.4	13.40	0.31	13.40
Calcium, Ca^{2+}	5410.8	270.0	6.16	135.0
Magnesium, Mg^{2+}	850.5	70.0	1.60	35.0

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Parameter	mg/L	meq/L	Equivalent fraction, %	mmol/L
Sodium + potassium (calculated)	44 431.0	1851.1	42.24	–
Dry residue, mg/L	149 358	–	–	–
pH / density, g/cm^3	7.4 / 1.055	–	–	–

Note: values in mg/L are taken from [4]; the other columns were calculated by the authors.

The sums of anions and cations (2191.1 meq/L) are fully balanced. The equivalent fractions of calcium and magnesium given in the primary source (3.1 and 4.2 %) do not correspond to the mass concentrations: the correct values are 6.16 and 1.60 %. Since rNa/rCl = 0.85 < 1 and (rCl – rNa)/rMg = 4.55 > 1, the water belongs to the calcium chloride type according to Sulin's classification (Fig. 1).

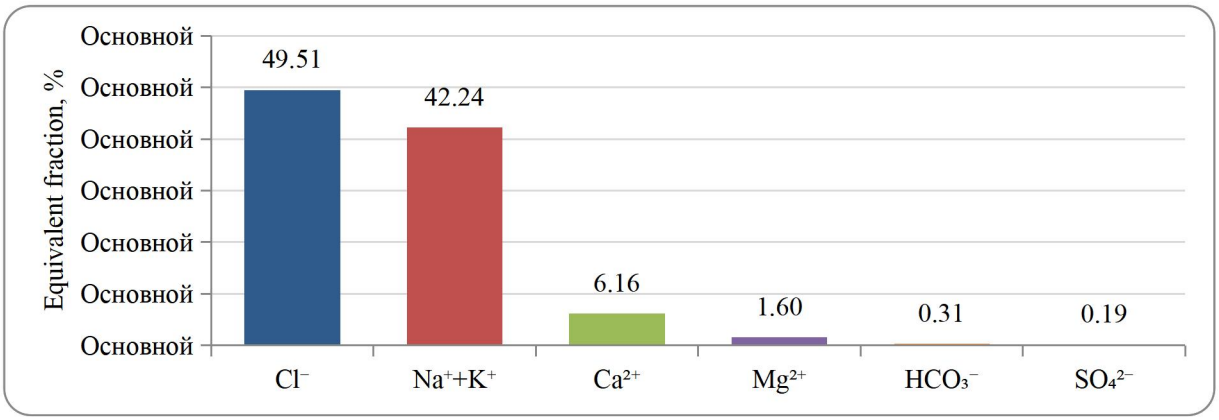


Fig. 1. Equivalent fractions of ions in the Chegara field formation water (authors' calculation)

The water is high in Ca^{2+} (135.0 mmol/L) and HCO_3^- (13.4 mmol/L) and low in SO_4^{2-} (4.13 mmol/L): when pressure drops and CO_2 is released, the main risk is carbonate scaling, and when the water mixes with sulfate-rich water, gypsum scaling. The GRDC-0621 inhibitor tested in this water provided 95 % protection against $CaCO_3$ scale at a dose of 30 g/t, whereas no effect was observed at 20, 40 and 50 g/t [4].

Stoichiometric reagent consumption. Since chelating agents form 1:1 complexes with metal ions and have large molar masses (K_4-EDTA – 444.60; K_5-DTPA – 583.80 g/mol), formula (2) gives 2.5–4.4 kg of pure substance per 1 kg of scale, whereas dissolving calcite requires only 0.73 kg/kg of pure HCl (4.86 kg of a 15 % solution) (Fig. 2). Hence, for carbonate scale a chelating dissolver is justified only where corrosion or equipment-sensitivity constraints apply. The K_2CO_3 converter, at a consumption of 0.59–1.02 kg/kg, turns sulfate into acid-soluble carbonate and reduces the required proportion of chelating agent.

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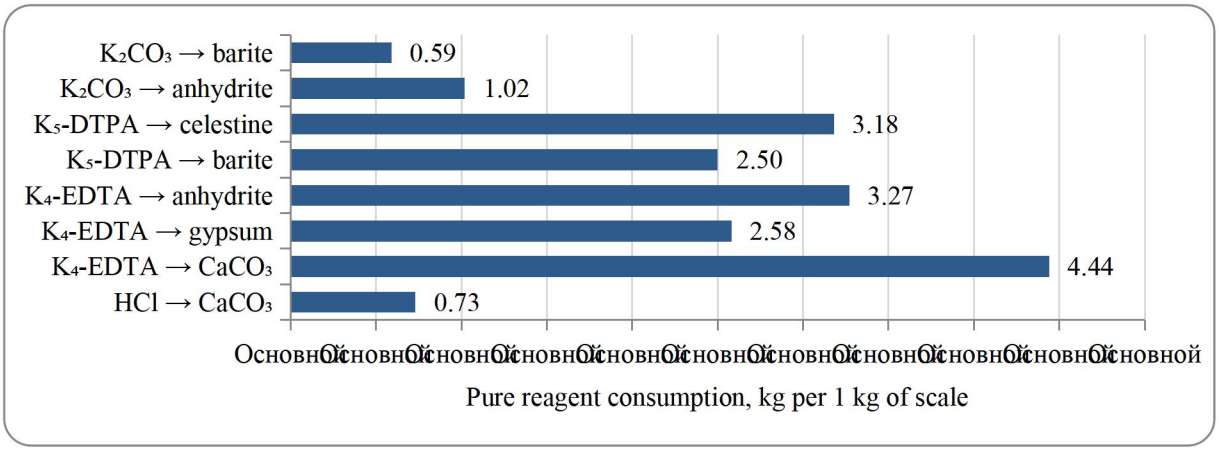


Fig. 2. Theoretical specific consumption of pure reagent in scale dissolution (authors' calculation)

Comparing theoretical and practical capacity makes it possible to estimate the reagent utilisation factor. A 0.5 M DTPA solution dissolved 47.2 g/L of barite at 100 °C in 7 h [15], while the theoretical capacity is 116.7 g/L, i.e. a utilisation factor of ≈40 %. The 20 % K_4-EDTA + 5 % K_2CO_3 system completely dissolved 5 % (≈50 g/L) of $CaSO_4$ [8]; assuming a density of 1 g/cm^3, K_4-EDTA is ≈0.45 mol/L, the theoretical capacity for anhydrite is ≈61 g/L and the utilisation factor is ≈82 %. Thus, the converter ensures fuller use of the chelating capacity.

Comparative efficiency of dissolver systems. The results of the selected experimental sources are summarised in Table 3.

Table 3

Comparative efficiency of salt deposit dissolvers

Dissolver system	Scale and conditions	Result	Source
20 % K_4-EDTA + 5 % K_2CO_3, pH 10.5	$CaSO_4$ 5 %; 93 °C; 3–6 h	100 %	[8]
20 % K_4-EDTA + 5 % K_2CO_3, pH 7	$CaSO_4$ 1–3 % / 5 %; 93 °C	100 % / 90.3 %	[8]
16 % MGDA, pH 10	Gypsum 2 g / 10 mL; 100 °C; 24 h	> 80 %	[12]
16 % GLDA / 15 % DTPA / 15 % HEDP	Same conditions	66 / 45 / 25 %	[12]
0.5 M DTPA (K), pH 11–12	Barite; 24 h	60 %	[10]
0.5 M DTPA + 5–7 % K_2CO_3 / KCl / HCOOK	Barite; 24 h	80–90 %	[10]
0.5 M DTPA	Barite; 100 °C; 7 h	47.2 g/L	[15]
EDTA	Gypsum / anhydrite	43 / 34 g/L	[3]
DTPA-based dissolver	Barite; 80 °C; 48 h (static)	Highest degree of	[16]

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Dissolver system	Scale and conditions	Result	Source
		dissolution	

The gypsum dissolution efficiency of chelating agents is compared in Fig. 3.

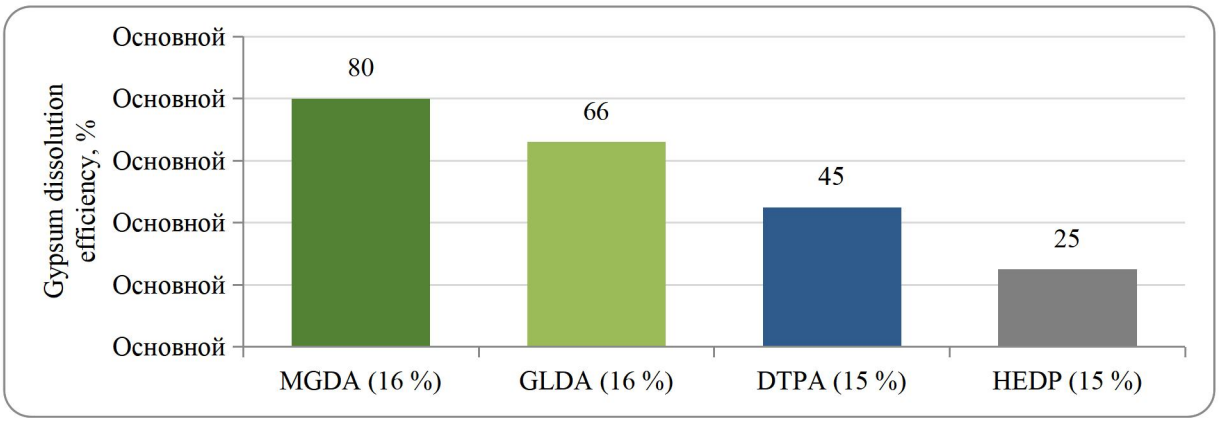


Fig. 3. Gypsum dissolution efficiency of chelating agents (100 °C, 24 h, optimal pH; based on data from [12])

In the K_4-EDTA + K_2CO_3 system [8], the efficiency was 100 % at 10 and 20 % K_4-EDTA and 84 % at 30 %; the optimal K_2CO_3 content was 5 % (87 % at 7 % and 90 % at 10 %). The effect of temperature was small: for the pH 7 dissolver, raising the temperature from 24 °C to 93 °C increased efficiency from 88.5 % to 90.3 %, while the pH 10.5 dissolver gave 100 % at both temperatures. The corrosion rate of steel coupons at 65 °C in the pH 10.5 dissolver was 2.83 kg/(m^2·year), well below the acceptable limit (9.76 kg/(m^2·year)) and the value in deionised water (18.2 kg/(m^2·year)). The dissolver remained stable at 120 °C for 3 days.

DISCUSSION

Carbonate scale is decomposed by hydrogen ions (4), while a chelating agent binds calcium into a soluble complex (5); the kinetics of calcite dissolution are controlled jointly by acid attack and chelation [17]:

$$CaCO_3 + 2HCl \rightarrow CaCl_2 + H_2O + CO_2\uparrow \quad (4)$$

$$CaSO_4 + Y^{4-} \rightarrow [CaY]^{2-} + SO_4^{2-} \quad (5)$$

$$CaSO_4 + K_2CO_3 \rightarrow CaCO_3 + K_2SO_4 \quad (6)$$

$$BaSO_4 + DTPA^{5-} \rightarrow [BaDTPA]^{3-} + SO_4^{2-} \quad (7)$$

In the conversion–chelating system (6), sulfate is bound as highly water-soluble K_2SO_4 (arcanite), while calcium passes into carbonate and forms a complex with EDTA; the product was confirmed by X-ray diffraction and is easily washed out with fresh water [8]. Compared with the two-stage "conversion + acid" method, single-step treatment shortens the reagent pumping time.

The superiority of DTPA for barite (7) is associated with its formation of a more stable complex with the large Ba^{2+} ion; it is also effective against strontium sulfate [13]. The role of catalysts is important: in the presence of 0.1–0.5 M oxalate, DTPA reached 80–90 % of saturation within 10

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minutes [18], and the addition of K_2CO_3, KCl or potassium formate raised the 24-hour dissolution degree from 60 % to 80–90 % [10]. Dynamic tests on formation samples showed that excessively high DTPA concentrations should be avoided [16].

The most important conclusion for local conditions concerns the water used to prepare the dissolver. Since the sum of Ca^{2+} and Mg^{2+} in the Chegara water is 170 mmol/L, a dissolver prepared with this water loses 0.17/0.5 = 34 % of the capacity of a 0.5 M chelating agent to dissolved cations before it reaches the scale. This estimate agrees with practical observations: the dissolving capacity of DTPA and EDTA dissolvers prepared with seawater decreased by about 20 % [19], whereas in low-hardness tap water the efficiency of the K_4-EDTA + K_2CO_3 system did not change [8]. In addition, magnesium and iron(III) ions in EDTA solution reduce gypsum solubility, and at low pH magnesium sulfate may reprecipitate [9]. Therefore, in fields with calcium chloride waters the dissolver should be prepared only with fresh or softened water.

From an environmental standpoint, more than 80 % of MGDA biodegrades within 14 days, whereas EDTA and DTPA practically do not degrade within 28 days [12]. GLDA is produced from a renewable raw material, sodium glutamate, and has low corrosivity [11]. In terms of local raw materials, the effectiveness of KCl as a catalyst [10] is of particular importance, since this salt is available in the country owing to the production of potash fertilisers; the feasibility of creating low-cost inhibitor compositions based on local chemical products has also been demonstrated [5].

Based on the results of the analysis, a process scheme for salt deposit dissolution was developed (Table 4).

Table 4

Proposed process scheme for salt deposit dissolution

Stage	Content	Control parameters
Diagnostics	Phase composition of the scale sample (X-ray diffraction, chemical analysis); ionic composition of formation water	Scale type; sum of Ca^{2+}+Mg^{2+}; water type
Formulation selection	Carbonate – 10–15 % HCl + corrosion inhibitor or GLDA; $CaSO_4$ – 10–20 % K_4-EDTA + 5 % K_2CO_3, pH 10.5; $BaSO_4$, $SrSO_4$ – 0.5 M K_5-DTPA + 5–7 % K_2CO_3 (KCl, HCOOK); where environmental requirements are strict – MGDA/GLDA	Theoretical consumption G according to Fig. 2 and safety factor
Solution preparation	Preparation with fresh or softened water	Water hardness; pH
Treatment	3–6 h for $CaSO_4$ (≈93 °C); 24–48 h for barite (80–100 °C)	Ca^{2+}/Ba^{2+} concentration in the solution
Flushing	Removal of reaction products (K_2SO_4) with	pH and composition of

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Stage	Content	Control parameters
	fresh water	returned fluid
Inhibitor protection	Optimisation of the field-specific inhibitor dose (GRDC-0621 – 30 g/t for Chegara)	Protection efficiency ≥ 90 %

Note: the scheme was compiled by the authors on the basis of the data in Tables 1–3 and Fig. 2.

A limitation of the study is that the compared data were obtained in different laboratories and at different scale-to-solution ratios; the need for standardised ranking of dissolvers has been pointed out previously [20]. Testing the proposed criteria on real scale samples from local fields is a task for further research.

CONCLUSIONS

1. The choice of dissolver is determined by the anionic composition of the scale: acid systems are preferable for carbonates (pure HCl consumption 0.73 kg/kg), whereas sulfates dissolve only in chelating and conversion systems.

2. The Chegara field water is of the calcium chloride type (rNa/rCl = 0.85) with 170 mmol/L of divalent cations; a 0.5 M chelating dissolver prepared with this water loses 34 % of its capacity, so the solution should be prepared with fresh or softened water.

3. For calcium sulfate, the 20 % K_4-EDTA + 5 % K_2CO_3 (pH 10.5) system completely dissolves up to 5 % of scale in 3–6 h (reagent utilisation ≈82 %, corrosion 3.4 times below the limit); for barite, adding K_2CO_3, KCl or potassium formate to DTPA increases the dissolution degree from 60 % to 80–90 %.

4. Biodegradable MGDA (> 80 %) and GLDA (66 %) outperform DTPA (45 %) and HEDP (25 %) in gypsum dissolution.

5. The proposed six-stage process scheme helps to systematise dissolver selection in local fields and to reduce dependence on imported reagents.

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