Review Paper

A critical review of self-diverting acid treatments applied to carbonate oil and gas reservoirs

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ABSTRACT

Carbonate reservoirs generally achieved relatively low primary resource recovery rates. It is therefore often necessary to clean those reservoirs up and/or stimulate them post drilling and later in their production life. A common and basic carbonate reservoir cleanup technique to remove contaminating material from the wellbore is acidizing. The efficiency of acid treatments is determined by many factors, including: the type and quantity of the acid used; the number of repeated treatments performed, heterogeneity of the reservoir, water cut of the reservoir fluids, and presence of idle zones and interlayers. Post-treatment production performance of such reservoirs frequently does not meet design expectations. There is therefore much scope to improve acidizing technologies and treatment designs to make them more reliable and effective. This review considers acid treatment technologies applied to carbonate reservoirs at the laboratory scale and in field-scale applications. The range of acid treatment techniques commonly applied are compared. Differences between specific acid treatments, such as foamed acids, acid emulsions, gelled and thickened acid systems, targeted acid treatments, and acid hydraulic fracturing are described in terms of the positive and negative influences they have on carbonate oil production rates and recovery. Opportunities to improve acid treatment techniques are identified, particularly those involving the deployment of nanoparticles (NPs). Due consideration is also given to the potential environmental impacts associated with carbonate reservoir acid treatment. Recommendations are made regarding the future research required to overcome the remaining challenges pertaining to acid treatment applications.

1. Introduction

The distribution of carbonate rocks constitutes approximately 20% of sedimentary rocks worldwide. Furthermore, carbonate reservoirs contain more than 70% of the world's oil and gas resources, including approximately 50% of the world's proven recoverable oil and gas resources (Lin et al., 2021; Xu et al., 2020). Currently, carbonate reservoirs hold about 60% of the world's proven plus probable oil reserves (Odintsova et al., 2021; Xiao et al., 2021). Moreover, there are about 400 oil and gas basins recognized worldwide in onshore and offshore locations (Krencker et al., 2020;

Xiao et al., 2021; Xu et al., 2020). Fig. 1 describes the distribution of recoverable reserves associated with the world's marine carbonate reservoirs, identifying that the most prolific carbonate oil and gas resources are located in the Middle East, the former Soviet Union, North America, and the Asia-Pacific area (Liu et al., 2017; Xu et al., 2020). These statistics highlight the importance of optimizing production from carbonate reservoirs in the global context of maximizing the recovery of the available oil resources.

Thus, the search for new and improved ways to intensify oil production from carbonate reservoirs is the most important challenge as carbonate reservoir rocks are characterized by multiple complexities. These complexities include: heterogeneous permeability and fracture distributions, the presence of flushed zones (Chacon and Pournik, 2022), and other complicating factors caused by the clogging of perforated zones (Martyushev et al., 2022;

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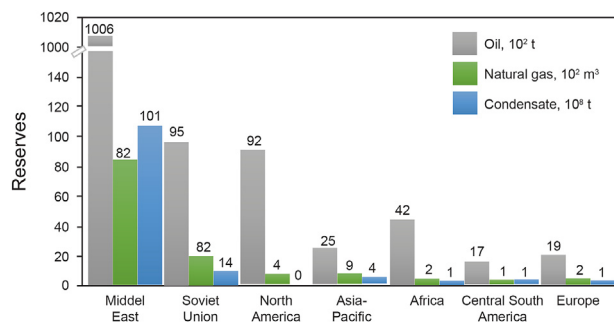E-mail address: davoodis@hw.tpu.ru (S. Davoodi).

Fig. 1. The world's confirmed and likely recoverable stocks of marine carbonates (Modified after Liu et al., 2017; Xu et al., 2020).

Martyushev and Vinogradov, 2021) and collimation of pore channels by rock particles during fluid movements into the wellbore (Yuan and Wood, 2018). These reservoir characteristics also lead to problems during well workovers related to the impacts of well-kill fluids. They also pose challenges during the late stage of development of producing reservoirs (El-Aziz Mohamed et al., 2021; Radwan et al., 2021). Currently, one of the most widespread and effective technologies aimed at increasing or restoring the productivity of production wells in carbonate reservoirs is acid treatment (Jafarpour et al., 2019; Yudin et al., 2021). The intensification of oil production from carbonate reservoirs with various acid compositions (AC) based on hydrochloric acid (HCl) has been deployed since 1895 (Trushin et al., 2019).

The essence of acid-injection treatments is to dissolve certain carbonate minerals and many colmatants (Jafarpour et al., 2019; Manakhayev et al., 2018). These treatments aim to artificially improve the permeability of the producing formations by increasing the number and size of drainage channels (Chacon and Pournik, 2022; Jafarpour et al., 2021). Furthermore, various technologies have been successfully applied to enhance acid-treatment efficiency in carbonate reservoirs, including the application of various acid compositions (Putilov et al., 2020; Xu et al., 2020). Depending on formation temperature, different concentrations of acids tend to be used. Such adjustments are aimed at slowing down the reaction rate of the acid composition with the reservoir formations, in order to increase the coverage of the treatment zone with acid.

Applying acids in dispersed systems (foams, hydrophobic emulsions, gels) or using acid-rock reaction rate retarders can help to achieve that objective (Czupski et al., 2020; Putilov et al., 2020). Hydrochloric acid's solubility and reactivity and low cost make it the most common reagent for acid treatment in carbonate reservoirs (Chacon and Pournik, 2022). However, there are two problems associated with hydrochloric acid deployment: reaction rate and corrosivity being too high. The acid mixture is often supplemented with organic acids to reduce the level of corrosivity. In addition, the lower solubility of organic acids reduces the irregularity of the acid reaction front in the rock, and reduces the risk of preferential wormholes diverting the acid away from the zones most in need of stimulation (Chacon and Pournik, 2022; Czupski et al., 2020; Gozel et al., 2021; Putilov et al., 2020; Xu et al., 2020).

The main reason for the low efficiency of most acid treatments of heterogeneous reservoirs is the irregular penetration of injected fluids into the formation, primarily flowing into the most permeable interlayers. These result in the permeability of already highly permeable interlayers increasing and the deepening of existing fractures in carbonate reservoirs. In turn, low-permeable interlayers are barely exposed to the injected acid (Gabzalilova et al., 2021). To prevent this, highly permeable interlayers need to be isolated temporarily with viscous material for the duration of acid injection (Evgenievich Folomeev et al., 2021).

One technique with the potential to increase zonal coverage during acid treatments of a reservoir involves self-diversion systems. These can be classified as mechanical self-diversion or chemical self-diverting acid technologies. Such systems create filtration resistance that hinders the injected acid from entering into the highly permeable zones of the target reservoir but rather diverts the injected stimulation fluids into the less permeable zones (Abdollahi et al., 2021; Ma et al., 2018; Mahdaviara et al., 2021; Zhao L. et al., 2020). However, mechanical and chemical diversion technologies work in different ways. Which type of diversion technology to deploy is typically determined by cost and specific reservoir properties such as porosity, permeability, and other properties (Chacon and Pournik, 2022; Garrouch and Jennings, 2017).

Ball sealants, packers, double packers, and coiled tubing are examples of mechanical diversion mechanisms (Abdel-Basset et al., 2021; Fragachan et al., 2015; Oberascher et al., 2012). Ball sealants are useful in vertical wells in target formations displaying large permeability contrasts, and they are preferentially positioned on the upper side of the wellbore in deviated and horizontal wells. Ball-sealant impacts tend to be complicated and are heavily influenced by a well's inclination angle, ball density, flow velocity, perforation orientation, and a formation's permeability contrasts. Setting packers and tandem packers required to deploy ball sealants can be costly and time-consuming. Moreover, it is not always practical or advisable set such mechanical devices in long horizontal or extended reach drilling (ERD) wells. Coiled tubing can be used effectively and cheaply to enhance acid transport via selective zonal treatments. If repeated acid injections do not show any positive effects, the coil can be quickly removed or repositioned. A major drawback to coiled tubing is that it restricts the pump rates that can be applied to inject the fluids, which tends to be a limitation to their use in deeper wells (Gomaa et al., 2011; Hosseinzadeh et al., 2017; Kalfayan and Martin, 2009).

The implementation of chemical self-diverting acid technologies is designed to provide a uniform intensification of the stimulation treatment across the entire productive interval of a carbonate reservoir. They act to minimize the degree of treatment contamination into already permeable zones. Compared to mechanical diversion technologies, chemical self-diverting acid treatments require fewer stages and less total injection volume, as they provide simultaneous acid stimulation and treatment diversion (Andreev, 2020; Quevedo et al., 2012; Zhao L. et al., 2020; Zhao M. et al., 2020; Zhao et al., 2022; Zhou et al., 2009; Zhu et al., 2022). The chemical self-diverting acid systems are typically based on foam (Evgenievich Folomeev et al., 2021; Yan et al., 2019), acid emulsions (Madyanova et al., 2012), gelated acid (Lynn and Nasr-El-Din, 2001; Sarmah et al., 2020; Yan et al., 2019), acid plus fiber treatments (Bukovac et al., 2012), and visco-elastic surfactants (VES) (Bulgakova et al., 2013; Hull Katherine et al., 2016; Mahdaviara et al., 2021).

It is important to understand all of the active mechanisms involved in chemical self-diverting acid systems and the impacts they have on a reservoir when implemented. Unfortunately, the overall reservoir impacts of chemical self-diverting acid systems are typically not well documented. This review focuses on studying and explaining the types of chemical self-diverting acid systems and their impact and importance as a new low-cost technology in improving oil production from carbonate rocks. Moreover, this review addresses technical difficulties involved in effectively developing carbonate oil reserves to optimize resource recovery. The following points are considered in detail:

- Carbonate formations and structures and applicable types of acid treatment;
- Types of self-diverting acid stimulation technologies available and their advantages, disadvantages, and mechanisms;
- Advanced self-diverting acid methods;
- Difficulties associated with acid stimulation techniques;
- Potential of nanoparticle (NP) additives to improve acid treatments;
- Future research and field-scale-testing requirements.

This review should also assist those new to the field in understanding the future research efforts required to further improve the existing technologies.

2. Types of carbonate reservoir rocks and their structures

Carbonate rocks and sediments include formations composed of 50% or more by carbonate minerals (Kontorovich et al., 2016). The most common carbonate minerals are compounds of calcite and dolomite, the crystals of which display trigonal syngony (Ahr, 2008; Kontorovich et al., 2016). Less commonly carbonate formations contain rhombic aragonite crystal forms. Naturally occurring mineral carbonates, other than those of calcium, such as magnesite ($MgCO_3$), rhodochrosite ($MnCO_3$) and smithsonite ($ZnCO_3$) are also present in some carbonate formations. As well as dolomite ($CaMg(CO_3)_2$), other mixed metal carbonate minerals also occur, such as ankerite ($CaFe(CO_3)_2$), and kutnagorite ($CaMn(CO_3)_2$) (Bagrintseva, 2015; Shen et al., 2015). Strontianite ($SrCO_3$) and cerussite ($PbCO_3$) exist with similar crystal forms to aragonite. These varied carbonate minerals are capable of isomorphism and form solid solutions with a wide miscibility (Bagrintseva, 2015; Chung-Cherng Lin, 1997; Shen et al., 2015).

Calcite is the most stable form of $CaCO_3$ over a wide range of temperatures and pressures. It frequently exists as pure $CaCO_3$ but can also contain the metals Fe, Mg, Mn. Most often, calcium is replaced by magnesium. Calcite with $MgCO_3$ content of more than 5% is referred to as high-magnesian calcite. Aragonite is a much less stable form of $CaCO_3$ and is found mainly in modern sediments of biogenic origin. Over time, it tends to spontaneously transform into calcite. Aragonite crystal lattices relatively rarely accommodate the Mg^{2+} ion up to 0.001%, whereas their strontium contents can be up to about 1%.

Dolomite bearing rocks are usually of chemogenic origin. The crystal lattice of dolomite is highly ordered and formed by one-for-one substitution of Ca atoms by Mg atoms. In dolomite, the place of Mg^{2+} is often taken by Fe^{2+} with the formation of a continuous series of solid solutions up to ankerite. Iron-enriched dolomite are more commonly associated with iron-rich sediments and ores rather than porous carbonate reservoirs. Rock-forming dolomite is generally characterized by low iron content (Ahr, 2008; Kontorovich et al., 2016; Shen et al., 2015). Depending on the predominance of $CaCO_3$ or $CaMg(CO_3)_2$, two main groups of carbonate oil and gas reservoir rocks are distinguished, limestones and dolomites, but often they occur together in a single rock formation.

Carbonate rocks containing substantial clay minerals exist as marls, which tend to have reduced porosity and permeability than pure carbonate rocks. The porosity of carbonate reservoir rocks tends to be lower on average than that of clastic reservoirs, however, their permeability can be higher due to fracturing and cavernous porosity. Some carbonate formations rich in fossil shells exhibit reservoir properties similar to those of sandstones (Bagrintseva, 2015; Shen et al., 2015; Xu et al., 2020).

Carbonate minerals of different composition and form react differently to acid treatment. There are two broad types of acid treatments: matrix and acid fracturing (AHF). Matrix acid treatments are injected at pressures below the formation's fracture pressure. They are primarily applied to repair formation damage caused by pore plugging or mineral deposition. In most matrix treatments acid is injected only about 1 m into the formation from a wellbore. On the other hand, AHF is a hydraulic fracturing operation in which acid is injected at higher pressures in one or more stages. In hydraulic fracturing, the depth of acid penetration into the formation is one to two orders of magnitude greater than for matrix acid treatments. Hydraulic fracturing requires fluids (water-based or oil-based gels) to be pumped into wellbore by powerful pumping units to achieve pressures higher than the fracturing pressure of the target formation, i.e., pressures must exceed the minimum mechanical stress of the target formation.

In some cases, the proppants are used to keep the induced formation fracture open (Aljawad et al., 2019; El-Aziz Mohamed et al., 2021; Yudin et al., 2021). However, AHF is only effective when the reaction rate between acid and the carbonate rocks is intense and rapidly executed. The acid compositions used typically include hydrochloric acid (HCl), combined with organic acids but vary according to formation conditions, particularly temperature. The acid composition used for AHF will vary based on the length and conductivity of the induced fractures. There are many factors that need to be taken into account when designing acid treatments (Aljawad et al., 2019; Daccord et al., 1989; El-Aziz Mohamed et al., 2021; Freed and Fogler, 1999; Yudin et al., 2021). Table 1 summarizes the criteria influencing acid treatment designs and applicable acid composition used to stimulate carbonate reservoirs.

2.1. Acid stimulation treatment conditions applied to carbonate reservoirs

Analysis of more than 650 acid treatments revealed that 73% of treatment failures were due to incorrect choice of acid-treatment technology, whereas 27% of treatment failures were due to incorrect choice of acid compositions, volumes and sequence of processing stages (Leong and Ben Mahmud, 2019). Among the possible reasons for the low efficiency of many carbonate reservoirs acidizing treatment is the preferential flow of injected acid into highly permeable interlayers with high water saturation. This leads to a premature increase in the water cut in a well's fluid production (Mohsen et al., 2019). Acid treatments are carried out to accomplish the following tasks: (1) reducing skin factor by removing mineral and organic deposits from the target formation zones; (2) increasing hydraulic conductivity of low-permeability zones within the formation; (3) aligning the flow profiles of production and injection wells. However, conventional acid treatments often fail to effectively achieve these primary objectives (Chang, 2022). By preferentially following the paths of least resistance, the injected acid leaves substantial volumes of low-permeability zones untreated. This ultimately leads to uneven treatment of the formation in area and depth (Fig. 2) (Chang, 2022; Leong and Ben Mahmud, 2019). Non-equalized interval treatment describes the irregularity of the injectivity profile during acid stimulation. The injectivity profile of specific reservoir zones is determined by: differences in zonal permeabilities; differences in zonal skin factor magnitudes; differences in rock reactivity from one zone to another; differences in zonal formation pressures; differences in the viscosity of formation fluids; the presence of fractures; a combination of these factors (Fig. 2) (Kalfayan and Martin, 2009; Perez et al., 2019).

Thus, based on these drawbacks and difficulties of using conventional acid treatments, it follows that one of the basic principles of improving the efficiency of acid treatments is the use of methods that reduce the filtration of working fluids into high-permeability interlayers. Such methods deflect the acid into low-permeability interlayers. It is, therefore, necessary to investigate and explain all

Table 1
Criteria influencing acid treatment designs and applicable acid compositions for use in carbonate reservoirs (Aljawad et al., 2019; Daccord et al., 1989; El-Aziz Mohamed et al., 2021; Fredd and Fogler, 1999; Yudin et al., 2021).

Types of acid treatment	Mechanism and definitions	Advantages	Disadvantages		Temperature limits for specific acid compositions	Types of carbonate reservoir rocks	Type of treatment-based acid
Acid hydraulic fracturing	This treatment uses acid as the fracturing fluid. The network of fractures and caverns created with acid and high pressure does not require proppant fixation. It differs from matrix acid treatment by applying a much higher volume of acid and injection pressure (higher than the rock fracture pressure).	Easy to perform and acid dissolves rock and increases channel conductivity. Wellhead pressure limitations are usually not an issue except when injecting a "cushion." Fracture conductivity can be higher than with proppants. Quicker well cleanup and well responses with limited problems associated with premature fracture shielding.	Fluid seepage into the formation is high due to the interaction of acid with the rock. Obtaining long acid etched fractures is impossible. Stable fracture conductivity at high closing pressures requires rapid acid distribution. Deep wells with high temperatures require more expensive organic and hydrochloric acid solutions. Corrosion inhibitors are always needed in soft carbonate rocks at high closing pressures.		$T \leq 149\ °C$, 15% HCl or 28% HCl; $149\ °C < T \leq 204\ °C$, 15% HCl or HCl–CH3COOH; $T > 204\ °C$, 10% HCl or HCl–CH3COOH.	Carbonate reservoirs that do not contain sediment-forming inclusions such as sulfates and iron compounds	Hydrochloric acid is preferred, but it is relatively rarely used without additives.
						Sulfate- and iron-containing carbonate reservoirs	It is preferable to treat with acetic and sulfamic acids.
Matrix acid treatments	Under the influence of matrix acid, voids, caverns and channels of erosion are formed in the rocks of the target formation zone. This results in increased permeability and, consequently, the productivity of oil / gas, or injectivity of injection wells.	Matrix acid treatment cleans up the perforated formations and the wellbore tubulars. It protects filters, tubing and other well elements from corrosion/precipitated products, such as waxes and mineral salts	Unable to control the direction of the pumped acid. High acid consumption. Limited benefits in conditions of high layer-by-layer heterogeneity of productive formations in terms of permeability. High reaction rate and high corrosiveness.	Recommendations for the applicable type of acid to use		Sulfate-containing carbonate reservoirs	Hydrochloric acid solutions with additives of calcium chloride or sodium chloride, potassium and magnesium sulfate, because they reduce the dissolution rate of sulfate-bearing minerals, i.e., prevent precipitation of gypsum or anhydrous calcium sulfate.
						Iron- containing carbonate reservoirs	Hydrochloric and acetic acid solutions preferred. Sedimentation is prevented by the inclusion of acetic or citric acid at mass fraction of 3%-5% and 2%-3%, respectively.

the techniques deployed as chemical self-diverting acid treatments with the aim of targeting acid distribution to specific zones within a carbonate reservoir. Chemical self-diverting acid treatment types are presented in Fig. 3, and Sections 3 and 4 explain the details of the methods.

3. Methods to self-diverting acid injected into carbonate reservoirs

3.1. Foamed-acid treatments

Foaming systems are typically two-phase with nitrogen or carbon dioxide as the dispersed phase. As an effective deflection method, they nevertheless require special control over the stability of the foam (Al-Shargabi et al., 2022b). The foam must have sufficient stability for a period of time before it is injected into the formation. Foam deployment times can be improved by employing coiled tubing units capable of rapidly delivering the acid-foam system to the formation (Chang, 2022).

The foamed-acid treatment mechanism involves slowing down the reaction velocity of acid and carbonate rock. By doing so it lengthens the exposure of formation zones to acid in the active state (Fig. 4). Once a foam is formed at a specific point in a reservoir it affects the rate of acid reaction with the surrounding rock. Foam dispersion involves H^+ mass transfer within the acidic water. H^+ is able to move and mix in all directions in basic oil/acid solutions due to Brownian displacement (Fig. 4a) (Hull Katherine et al., 2016). However, the pressure gradient in the aqueous phase forces the solution to move into the surrounding sphere in a limited way. CO_2 develops at the edge of the expanding sphere of foamed acid in the form of reaction bubbles, which act to decrease the capillary pressure. As the capillary effects change beyond certain limiting values, the CO_2 will fracture the bubbles and develop in the reservoir as supercritical CO_2 foam. At that point, the acid solutions become acid foams (Fig. 4b) (Hendrickson et al., 1992; Hull Katherine et al., 2016).

This makes it feasible to adequately control the flow rates of

injected foam fluid into reservoir layers with varying permeability. The employment of this property of foam fluids can boost acid penetration into formations with poor permeability thereby enhancing acidification productivity (Fig. 4c) (Yan et al., 2019). However, the action of a foaming agent and a stabilizer of acidic composition produces a gas-foam fluid, with a better chance of diverting the effects of the acid to the formation matrix and away from the high permeability pathways. Essentially the processes involved in the reservoir relate to the interface effects of a complex and dynamic gas/liquid/solid, multiphase, fluid system (Evgenievich Folomeev et al., 2021; Yan et al., 2019). CO_2 is more commonly used as a base agent than N_2 gas because CO_2 gas has higher density, higher diffusion coefficient, higher solubility in oil and water, and poorer interfacial characteristics. However, CO_2 has a higher corrosion effect on surface and subsurface equipment, and therefore requires the addition of a corrosion inhibitor, increasing its cost.

Acid foams also contribute to increasing the acidizing treatment coverage within the reservoir, as they have low density ($300-800\ kg/m^3$) (Mills, 2007), serving to increase their viscosity and strength. Foamed-acid treatments can be used to stimulate productive formations that are composed of carbonate rocks or calcareous sandstones, particularly sandstones with carbonate cements (Chacon and Pournik, 2022; Evgenievich Folomeev et al., 2021).

Foamed acid treatments have a number of advantages over conventional acid treatments (Letichevskiy et al., 2017). Much less fluid is pumped into the formation, which reduces the risk of contamination/formation damage. The foamed acid is more viscous than simple acidizing fluids, which ensures deeper acid penetration into the formation by reducing the reaction rate of the foam acid with the rock. Treated wells tend to be easier to clean up due to the low density of acid-foam composition. Foamed acid tends to result in more intensive flushing of contaminants from the target formation zones, as solid particles of contaminants are more easily suspended in the foam (Abdollahi et al., 2021; Letichevskiy et al., 2017; Zhao L. et al., 2020). The quality of the foam is determined
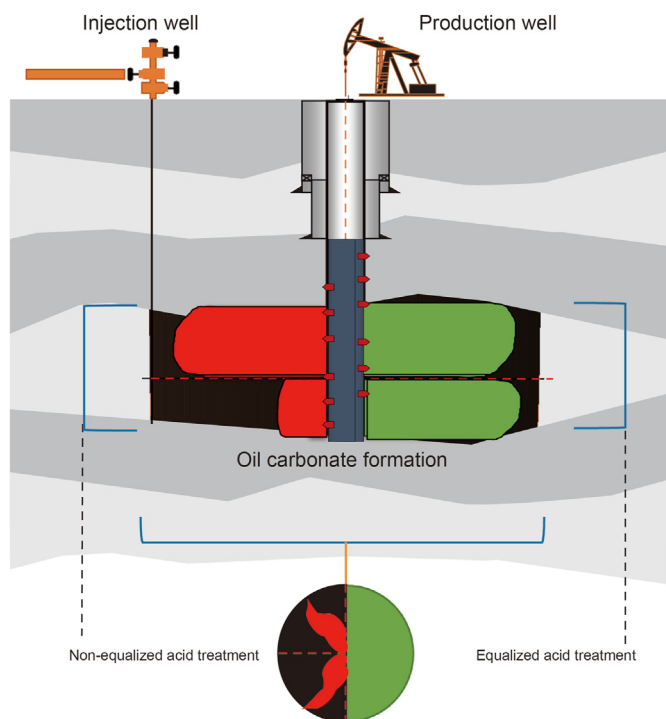
Fig. 2. Equalizing acid treatment by controlling injection rates into carbonate reservoir zones.
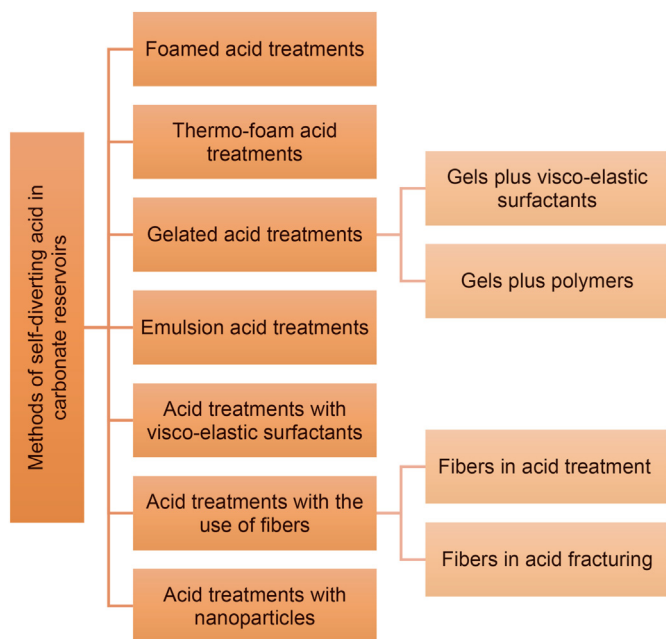

Fig. 3. Types of acid treatments applied to carbonate reservoirs.

by the volume content of the gas in it. The gas phase is generated from nitrogen or carbon dioxide booster plants. Foam is prepared at the surface by mixing gas and acid treated with a foaming agent. Foam quality is regulated by adjusting the flow rates of the liquid and gas phases (Letichevskiy et al., 2017).

The Kuleshovskoye field (Samara, Russia) provides an illustrative example of the effectiveness of foamed-acid treatments (Letichevskiy et al., 2017). The main advantage of foam as a
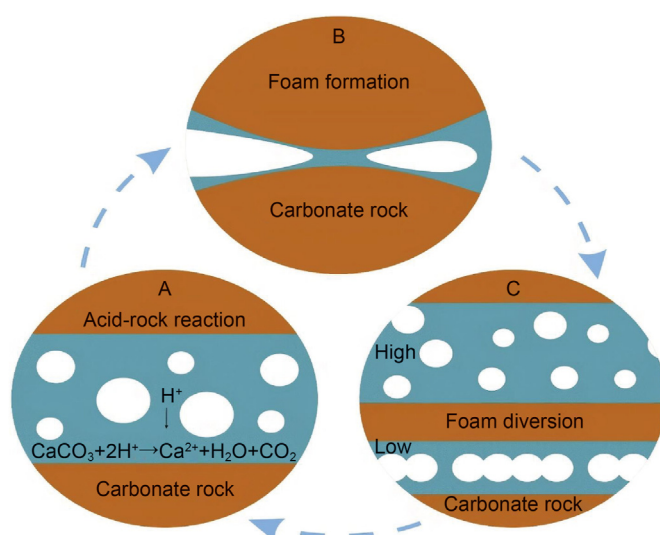

Fig. 4. Schematic illustration of a foamed-acid treatment in a carbonate reservoir (reproduced after Yan et al., 2019).

deflecting agent (Fig. 5), unlike polymer compositions, is its quick and effective removal from the formation, flushed by nitrogen. This is especially important in wells with low formation pressure. The main steps of nitrogen-fluid treatment are shown in Fig. 5b. Firstly, pre-cleaning is performed to remove hydrocarbons from the target zone. This is of great importance, as the foam is destroyed when coming into contact with hydrocarbons. Next, a buffer stage (linear gel with a surfactant-based foaming agent) is injected to displace solvent from the bottomhole zone and prevent its contact with the foam. The foam composition then enters the formation and preferentially blocks the high-permeability zones. After the foam enters the target zone, injection is briefly halted to improve foam formation. After the foam stabilizes, the main treatment fluid containing the foam agent is then injected (Evgenievich Folomeev et al., 2021; Garrouch and Jennings, 2017; Letichevskiy et al., 2017). The results demonstrated that the use of a nitrogen-foam diverter achieved an effective treatment of irregular intervals with interlayered water-saturated interlayers and some poorly cementing permeable zones. The productivity factor showed a 38% increase, and average normalized cumulative production increased by 58.6% over the six months following the sequential foamed-acid treatment (Fig. 5b) compared to wells stimulated with standard acid treatments (Letichevskiy et al., 2017). Moreover, the success of field applications using foamed-acid treatments mainly depends on geological factors (e.g., choice of the candidate well, the porosity of the reservoir, reservoir type, clay content, etc.), and technological features (e.g., volumes of pumped acid reagents, foam-forming compositions, foam multiplicity, aeration degree, alternation of foam and acid portions pumped).

Foamed fluids have demonstrated their ability to achieve uniform reservoir distribution due to their viscosity and their ability to break and reform in dynamic flow conditions. However, foamed-based, acid-diversion systems do not always remain stable for long periods prior to being injected. Foam stability is influenced by the type and concentration of surfactants involved, which is usually in the range of 0.1%–0.5% of solution volume. Additionally, mixing energy and foam quality influence a foam's stability. Foam quality is influenced by the average size of the bubbles, the homogeneity of the bubble size, and viscosity.

Table 2 describes the performance of variously configured foamed-acid diversion treatments.

3.2. Thermo-foam acid treatments

Thermo-foam acid treatment (TFAT) is a physicochemical stimulation technique suitable for carbonate formations - limestones, dolomites, and dolomitized limestones. TFAT is designed to restore or improve the reservoir properties of the bottomhole formation zone carbonate rocks. Thermal acidizing mechanism refers to the process of treating target formations with hot foamed acid. The acid is heated directly downhole adjacent to the injection zone to maximize the thermal effect. After thermal acid treatment, well cleanup with a two-phase foam ensures the removal of reaction products and materials that potentially contaminate the invaded portion of the target formation zone (Fig. 6) (Folomeev et al., 2020; Goenka et al., 2014).

The heat-generating process involved releases enormous amounts of heat, gases, and hot foamed acid. The heat melts high-molecular-weight oil molecules, cleans oil sheen (residual oil) from the rock surface, and accelerates the breakdown of the HCl. That breakdown helps to prevent premature deposition of dispersed colmatants, as well as reducing the rate of corrosion of underground equipment. The foam generated serves as a diverter for the next pulse of active acid injected, preventing unwanted stimulation of high-permeability interlayers and fractures. The surfactants in the acid solution improve its ability to penetrate pores and microfractures. Physical modeling of the thermal-foam acid treatment has been successfully conducted explaining its behavior (Crowe C et al., 1992; Goenka et al., 2014).

Two heat- and gas-generating solutions were combined with an activator to estimate the potential temperature of a heat-generating reaction (Folomeev et al., 2020). The activator was a highly concentrated HCl solution. The reagents were mixed in a stoichiometric ratio. During the experiment, the temperature at the reaction peak was 70 °C. The formation of foam as a result of mixing the ingredients was confirmed. A fivefold increase in the initial amount of solution was observed (Fig. 7). After pumping the acid through 1.6 pore volumes of the sample without replacing it, a breakthrough occurred. Heating provided a significant increase in permeability after exposure to HCl solution under conditions of wax blockage. The use of chemical heating was shown to increase the efficiency of acid treatment in the conditions of the formations tested (Folomeev et al., 2020).

A novel EIOR method involved an in situ thermo-foam system (Fig. 8), involving the preparation of a foam system in the pore space of the reservoir, by alternately pumping surfactant solution and gas into it (Kozhin et al., 2021). The thermal-foam system used in the experiments is generated by a combination of CO_2 and N_2 gases. As a result of thermal and gas chemical effects a local zone of high temperature and pressure is formed. This is associated with a decrease in viscosity, an increase in surfactant mobility, an increase in target-zone permeability due to the removal of colmatants and the formation of a network of micro-fractures in the rock formation. Advantages of this in-situ, thermo-foam acid system are that it can be applied at any depth in a wellbore, it is relatively cheap to implement compared to other thermal-acid methods, and is highly efficient, because it generates thermal energy in the specific area of interest within the target zone (Kozhin et al., 2021).

Therefore, compared to standard foamed-acid treatments, the TFAT offers the following advantages: stable foam, allowing for increased acid treatment coverage and slower acid-rock reaction rate; no precipitation on contact with oil; temporary heating of
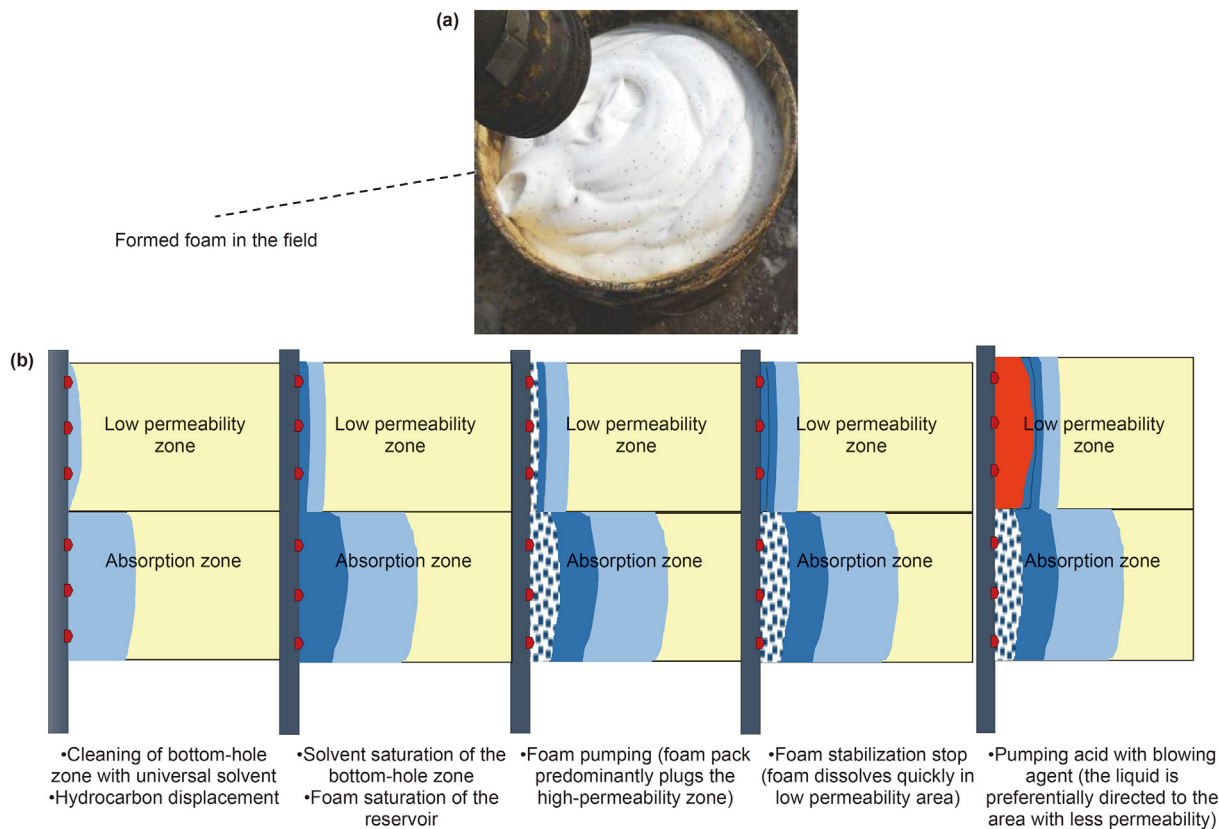

(a)

Formed foam in the field

(b)

| Low permeability zone | Low permeability zone | Low permeability zone | Low permeability zone | Low permeability zone |
| Absorption zone | Absorption zone | Absorption zone | Absorption zone | Absorption zone |

•Cleaning of bottom-hole zone with universal solvent
•Hydrocarbon displacement

•Solvent saturation of the bottom-hole zone
•Foam saturation of the reservoir

•Foam pumping (foam pack predominantly plugs the high-permeability zone)

•Foam stabilization stop (foam dissolves quickly in low permeability area)

•Pumping acid with blowing agent (the liquid is preferentially directed to the area with less permeability)

Fig. 5. Field examples of foamed-acid treatment: (**a**) nitrogen foam in the field; and (**b**) operating principles and sequence of the nitrogen-foam diverter (modified after Letichevskiy et al., 2017).

formation to promote the melting asphaltene-resin-paraffin solid deposits.

Table 3 describes the applications of thermo-foam acid treatments.

3.3. Emulsion acid treatments

Emulsions are colloidal systems composed of two immiscible liquids in which one liquid (dispersed phase) is dispersed in another continuous liquid (dispersion medium). Usually, these phases are water (aqueous solutions) and an organic liquid, often loosely referred to as an "oil" (Langevin et al., 2004). Emulsions are divided into two basic types, based on which fluid is distributed in which: oil-in-water (direct emulsions), and water-in-oil (inverted emulsions). The rheology of an emulsion is influenced by multiple factors that all interact with each other (Adil and Onaizi, 2022; Psillakis, 2019). Emulsifiers can be: 1) surfactants or colloidal electrolytes; 2) high molecular weight compounds (HMB); 3) highly dispersed powders, in particular hydrophilic substances (e.g., alkali metal soaps, gelatin, albumin, tannin, protein, chalk, gypsum, clay) that generate oil-in-water-type (O/W) emulsions, and hydrophobic substances (e.g., polyvalent metal soaps, lanolin, rubber, ceresin, paraffin, soot, carbon black) that generate water-in-oil-type (W/O) emulsions (McClements and Jafari, 2018). Emulsifiers can also be broadly classified as bipolar, organic molecules (surfactants in general) and solids (Fig. 9). Particulate matter can be either water- or oil-wetting; it self-organizes at the interface between the continuous and dispersed phases, forming a steric phase barrier to avoid coalescence. A Pickering emulsion is a solid-stabilized emulsion. However, surfactants have the ability to dissolve in both phases. They have hydrophobic and hydrophilic branches with oil and water affinity, respectively, and produce an interfacial film at the interface between the two phases, reducing interfacial tension (IFT). The consequent decrease in IFT leads to improved droplet dispersion and, as a result, a greater proclivity to emulsify (Adewunmi et al., 2022; Chevalier and Bolzinger, 2013). For acid treatment, acid-in-oil systems are used, corresponding to W/O emulsions. The concentration of the dispersed acid phase is about 70-75 %, which makes such systems highly concentrated (Adewunmi et al., 2022). The acid-in-oil mechanism acts to form a barrier between dispersed acid droplets and the reactive surfaces (either tubulars or mineral surfaces within the reservoir formation). In the case of equipment and tubing, this reduces corrosive attack, while in the case of carbonate reservoirs, it significantly reduces the rate of acid-mineral-grain interactions and consequently increases the depth of treatment. This effect is known as "retarded acid", and in addition to emulsions, it usually includes a variety of "thickened" and "inhibited" acids, where the reduction of the interaction rate is achieved not by forming a barrier but by reducing the diffusion rate (creating diffusion limitations). In addition to the direct "slowing down" of acid-in-oil systems, they have some additional positive properties of emulsion systems. When contacting a hydrocarbon fluid, a decrease in viscosity occurs (reducing the volume fraction of the dispersed phase and modifying the viscosity of the dispersed medium). On the other hand, when contacting an aqueous fluid (increasing the dispersed phase by incrementing its volume fraction) an increase in viscosity occurs. Some thickening also occurs as the calcium cation content increases (Fig. 9) (Adewunmi et al., 2022; Aldakkan et al., 2018; Sayed and Nasr-El-Din, 2011).

According to Ahmed et al. (2018), a new emulsified acid, waste oil system showed good potential as a fluid for intensifying oil production. A smaller pore volume of breakthrough (PVBT) acid is achieved in comparison to traditional diesel emulsified acid and

Table 2
Performances of variously configured foamed-acid diverting treatments.

Type of gas	Concentration	Work conditions	Findings	Ref.
CO$_2$ in a supercritical state	36.8% HCl /0.5% sodium dodecyl benzene sulfonate (SDBS)/0.15% carboxymethyl cellulose (CMC)	0.5 % SDBS (foam agent), 0.15 % CMC (stabilizer) at laboratory conditions of 20—60 °C, 7—10 MPa.	The flow ratios of injected foam fluids into different permeability zones can be adjusted. The utilization of this property of foam fluids to boost acid injection to medium and low permeability formations in order to improve acidification efficiency is a potential benefit of foam acidification treatments. In oil-water reservoirs, the process of selective diversion is the same as that for variable permeability layers. The continuous circulation process is initiated by acid-rock contact, which leads to foam creation and subsequently the diversion of acidic fluids. This results in deeper penetration of the foamed acid into the target zones.	Yan et al. (2019)
Nitrogen (N$_2$)	15 wt% hydrochloric acid/6 wt% Gemini quaternary ammonium salt cationic surfactant (GQA)/ 1 wt% corrosion inhibitor/1 wt% ferric ion stabilizer.	The optimal acid injection rate was 1—2 mL/min, as the injection rate increased, the wormhole morphology changed from conical-shaped wormholes to ramified wormholes. The quality of foam is approximately 90% of stability, as demonstrated by field applications under ambient conditions.	When the contrast of permeability among the zones was close to ten, foamed-VES acid may achieve uniform acid dispersion and overcome the restriction of ordinary VES acid. When the contrast of permeability exceeded 20, the foamed-VES acid was rendered ineffective. Furthermore, the low-permeability core sample tested had less than 30% wormhole penetration, showing that the bulk of low-permeability core sections were not activated by acid. The flow-rate distribution curve demonstrated that the foamed-VES acid could not achieve a diversion when the initial permeability contrast was increased to 20.1 (84.5:4.2) because the flow rate in the high-permeability core was always greater than that in the low-permeability core throughout the acidizing experiment.	Zhang et al. (2021)

pure HCl. The diesel emulsion performed better at lower injection rates, whereas the waste oil emulsion performed better at higher injection rates. This suggests that while diesel emulsion is superior in terms of stimulation efficiency, emulsified acid from waste oil may also be suitable for acid stimulation due to its low cost and adequate performance. Computed tomography investigations reveal that emulsified acid from waste oil may produce narrow, branchless, and deep wormholes, which are desirable properties for fluid-flow stimulation. Fig. 10 illustrates that for waste oil emulsified acid, the optimal PVBT was 5 cm^3/min, while for normal diesel emulsified acid optimal PVBT was 2 cm^3/min. At 2 cm^3/min, the novel emulsified acid exhibited essentially identical PVBT to diesel emulsion, indicating that the emulsion may attain the same performance as diesel (Ahmed et al., 2018).

Sidaoui et al. (2018) prepared emulsified acids using cationic emulsifiers, and performed a stability evaluation at the high temperature of 135 °C immediately after the emulsified acid had been prepared with a 28 wt % of HCl concentration and a 70:30 acid-to-oil ratio. The experiments were carried out with this high concentration; each agent's concentration being lowered by one-third. The results demonstrated that the most stable emulsified acid was obtained with no iron control agent and a low initial chelating agent concentration. Furthermore, varying quantities of iron control agents produce a noticeable difference, particularly after 2 h of heating. The most favorable quantity of the chelating agent was found to be the initial concentration. Additional increases in concentration showed no distinctive trend. Moreover, raising or lowering the quantity to four-thirds or two-thirds of the original concentration resulted in the same weight loss response (Sidaoui et al., 2018). Yousufi et al. (2019) prepared an emulsified acid using a silver nonionic emulsifier. It was able to form an efficient type of wormhole through an acid treatment core flow test. Although the emulsified acid was stable for more than 5 h at a temperature of
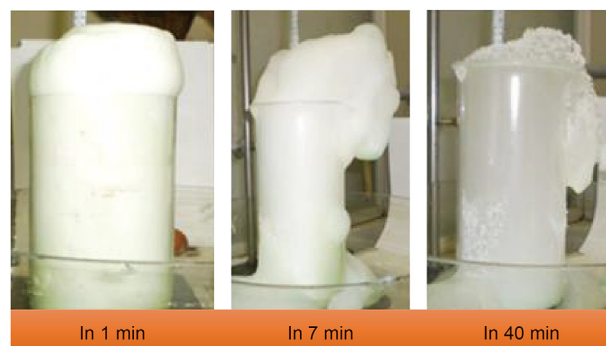

Fig. 7. The overall appearance of the reagents during the process of forming a thermo-foam acid (modified after Folomeev et al., 2020).

70 °C, it showed low stability for less than 9 h at room temperature. These studies suggest that while emulsified acid can act to reduce the reactivity of HCl and perform better in acid stimulations than HCl alone, their field deployment is problematic because they do not possess sufficient stability at room temperature or high temperature. Therefore, it is necessary to produce emulsified acids with sufficient stability at both low- and high-temperature conditions for field application (Yousufi et al., 2019). The stability of emulsions can be improved by mixing two or more different emulsifiers. For example, a mixture of nonionic emulsifiers and cationic emulsifiers was used experimentally to increase the stability of niosomes (Roque et al., 2020). The stability of the niosome, which had previously been maintained at room temperature for up to 7 days, was increased to 32 days when the mixed emulsifier was used (Roque et al., 2020). The use of acid-in-oil emulsions together with visco-
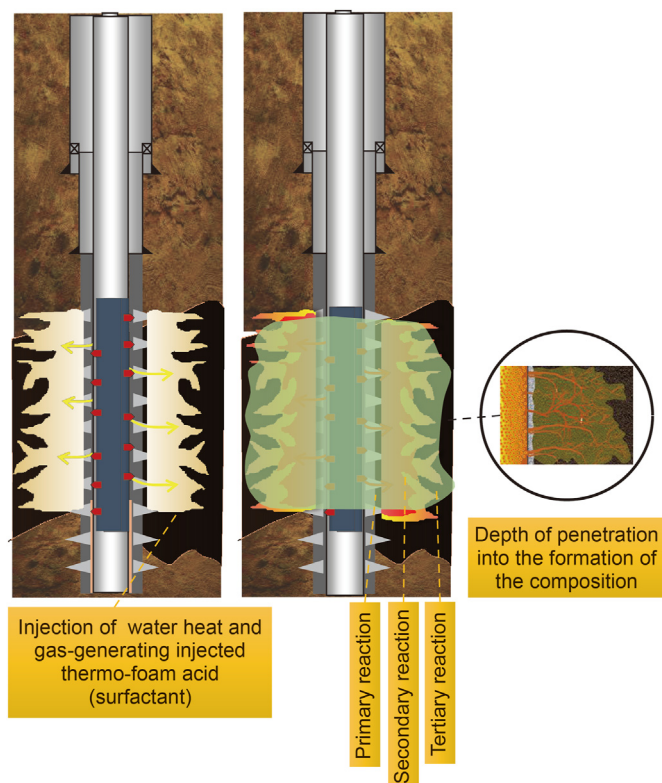

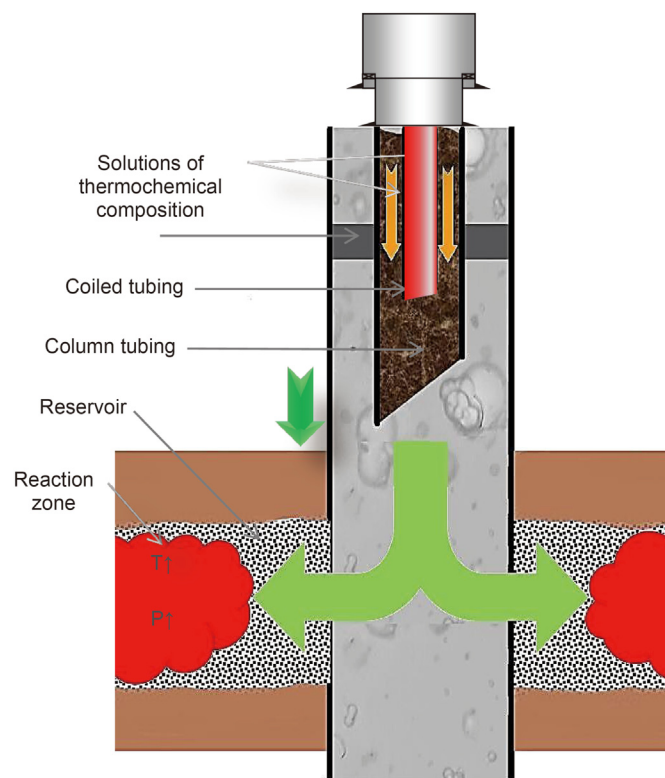**Fig. 6.** Mechanisms associated with thermo-foam acid well treatments.

Fig. 8. Mechanisms associated with *in-situ* thermo-foam well treatments (modified after Kozhin et al., 2021).

elastic-surfactant (VES) solutions as a diverter can also provide emulsion stability benefits. For instance, the use of a high-temperature emulsified acid and a high-temperature VES successfully stimulated an entire interval with a significantly higher productivity index (Madyanova et al., 2012). The diverting agent plays a very important role when stimulating thick formation intervals to ensure good zonal coverage. The post-stimulation skin factor value in the pressure recovery test was 3.3, and production log analysis revealed complete and uniform zonal coverage, with the upper zones contributing 53% of total well production and the lower zones accounting for 47% (Madyanova et al., 2012). The use of an emulsion acid composition to treatment formations has several advantages. It increases the penetration depth of the injected fluid and avoids the formation of precipitates. Highly permeable water-saturated interlayers tend to be blocked more easily and filtration flows redistributed. The treatment coverage of the formation is increased by these impacts. However, the emulsion compositions typically used do have some disadvantages: a relatively small depth of penetration into the reservoir; low viscosity; incompatibility

with oil in the presence of iron (III) ions; and poor efficiency in dispersing asphalt-resin-paraffin deposits contained within the oil-saturated reservoir zones.

Table 4 describes compositions of the most commonly used emulsified acids for carbonate reservoir stimulation.

Table 5 describes the carbonate core sample specifications and the experimental conditions used to evaluate the performance of emulsified acids in experimental tests.

Table 6 describes the results of specific laboratory tests of emulsified acid stimulation applied to carbonate cores.

3.4. Gelated acid treatments

When gelling agents are introduced to acids to generate a viscosified acid, a gelled acid develops at the surface. The injection pressure in this procedure is higher than that used in situ gelled acid, because of the larger pressure loss in the well column. Gelated acids display excellent performance in oil reservoirs at temperatures below 120°C (Abdollahi et al., 2021; AlOtaibi et al., 2020).

Table 3
Description of field-scale and laboratory-scale applications of thermo-foam acid treatments.

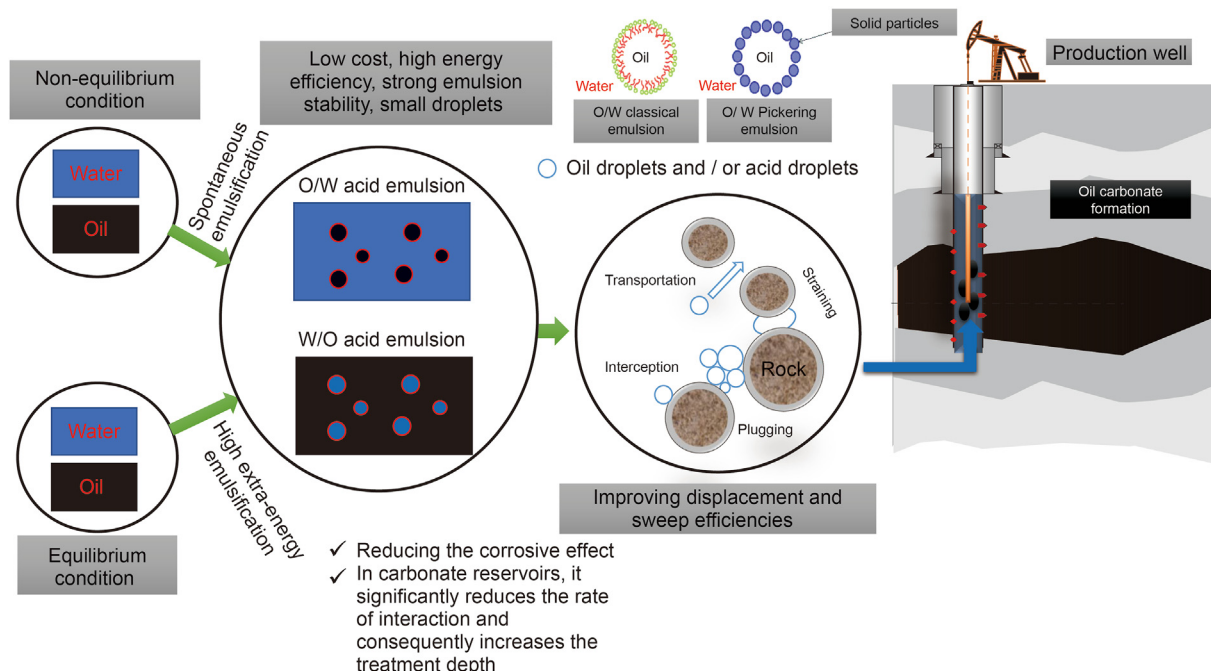| Reservoir properties	Chemical composition	Findings	Ref.
Podolski formation (Russia). The reservoir contains oil with a density of 861 kg/m^3, viscosity −10.9 mPa·s The oil is high in sulfur (2.5%), paraffin-base (2.4%), low-resin (14.9%). The reservoir temperature averages 20 °C, the weighted average reservoir pressure is 8.2 MPa and the average permeability is 19 mD.	Solution #1 involved carbamide and ammonium chloride. Solution #2 — carbamide and potassium nitrite. 12% HCl was used as an initiator, 2000 ppm (for Solution #1) and 5000 ppm (for Solution #2). An iron stabilizer and sodium hydroxide were added to the residual concentration of HCl. A mixture of surfactants is added to Solution #2 plus an activator to generate foam. The injection was performed with a set packer.	During the pilot technology test, the following injection data were recorded: bottomhole pressure and temperature, wellhead pressure, and the amount of injected reagents. An increase in bottomhole temperature and pressure signaled the start of a heat-generating reaction and the penetration of most of the acid solution into the formation. The increase in temperature measured by downhole sensors was consistent with laboratory test results. During injection, a significant increase in bottomhole temperature was observed in two of the three wells. This was due to reactions in the tubing below the packer, with the majority of acid involved in interaction with agents in the reservoir, indicating the start of a heat-generating reaction in the perforated interval. To avoid too early an onset of the reaction, the preparations must be injected continuously.	Folomeev et al. (2020)
Famennian formation (Russia). The reservoir contains oil of density 881 kg/m^3, viscosity −36.2 mPa·s. The oil is high in sulfur (3.4%), paraffin-base (2.6%), low-resin (21.9%). The reservoir temperature averages 31 °C, the weighted average reservoir pressure is 13.6 MPa and the average permeability is 3 mD.		The effectiveness of the technology was evaluated. The increase in oil flow rate per well averaged 2.3 t/day. During the 7 months following the treatment, the additional oil production was 858 tons, and that benefit persisted long after the treatment was conducted.	
The average permeability of the core model is 459×10^{-3} μm^2. The average porosity of the core model is 19.3%. Formation water in the for-core model is a NaCl solution used for oil displacement has a density of 1.044 g/cm^3. Oil saturating the core model has a density of 0.932 g/cm^3 and viscosity of 312 mm^2/s.	Without surfactant Solution #1 consists of HNH$_2$SO$_3$ - 30%, NaOH - 12%. Solution #2 is the same but with NaNO$_2$ - 21%.	Thermal gas-generating compositions based on sodium salts of sulfamic and nitric acids were evaluated in these laboratory tests. The dependence of the value of interfacial tension (IFT) on the mineral composition of water and temperature of the system "hydrocarbon-surfactant solution" was obtained experimentally. Increasing the temperature of the inorganic salt solution with surfactant led to an increase in surface activity, which also contributed to an improvement in the oil-washing ability of the solutions. Filtration	Kozhin et al. (2021)
The average permeability of the core model is 428×10^{-3} μm^2. The average porosity of the core model is 18.8%. Formation water saturating the core model for oil displacement is NaCl solution of density 1.044 g/cm^3.	With surfactant Solution #1 HNH$_2$SO$_3$ - 30%, NaOH - 12%. Solution #2 NaNO$_2$ - 21%, IF9-12 0.2%.	experiments showed that injection into the pore space of the reservoir model with residual oil saturation of thermal gas-generating composition of one pore volume led to an increase in the oil displacement ratio of up to 18%. Oil displacement after injection into the pore space of the model and resumption of water filtration has a "piston effect" causing subsequent gas breakthrough due to its high mobility. The formation of a dense foam system in the pore space of the reservoir, required a higher injection pressure, to prevent early breakthrough of the gas phase. The experiments confirmed that this contributed to an increase in the degree of extraction of high-viscosity oil.	

Fig. 9. Emulsion acid treatment mechanisms

However, when acidifying gas wells, the gel tends to damage the formations, so are best avoided. On the other hand, *in-situ* gelled acids and viscoelastic diverters are considered suitable for most types of reservoirs (Abdollahi et al., 2021). For heterogeneous carbonate reservoirs, characterized by differences in permeability, acid diversion is necessary to increase the effectiveness of acid treatments. *In-situ* gelled acid systems can be used for this purpose to reduce leakage rates during acid fracture stimulation (Arthur et al., 2009; Bulgakova et al., 2013; Sadeghnejad et al., 2022). In situ gelled acids are polymer-based systems consisting of an acid solution (mainly HCl), corrosion inhibitor mixed with a polymer, crosslinking agent, buffer, and destructor (AlOtaibi et al., 2020; Arthur et al., 2009; Kalgaonkar et al., 2022; Liu et al., 2021; Sadeghnejad et al., 2022).

3.4.1. In-situ gelled acids with visco-elastic surfactants

The use of self-diverting acid compositions based on visco-elastic surfactants (VES) is the most effective method for stimulating oil production from carbonate reservoirs. The advantage of VES-based acids is their ability to build up viscoelastic properties for acid diversion during the well treatment, and subsequently break down after the acid is neutralized. VES-surfactant solutions are often created by adding certain reagents to concentrated surfactant solutions, which frequently contain long-chain amphoteric and quaternary ammonium salts, such as cetyltrimethylammonium bromide. Many common chemicals and secondary surfactants, depending on their degree of ionization, can be added to surfactant solutions to increase their viscoelasticity and stability (Cetin and Nasr-El-Din, 2017).

There are many surfactant types that are capable of forming viscoelastic solutions. Those used, or tested for use, to stimulate oil reservoirs include: zwitterionic/amphoteric surfactants like betaine-based surfactants such as oleylamidopropylbetaine (Xie et al., 2022a). cationic surfactants such as erucilmethyl-bis-(2-hydroxyethyl) ammonium chloride4-erucamidopropyl-1,1,1-trimethylammonium chloride; anionic surfactants like sulfosuccinates (Leong and Ben Mahmud, 2019); aminoxides and

amidoaminoxides like tallic acid dimethylaminopropylamide oxide; ethoxylated fatty amines; Ionic pairs of cationic surfactants and anionic surfactants, such as sodium oleic acid, etc. (Kelland, 2014). Therefore, nonionic surfactants, unlike cationic surfactants, are ineffective at removing residual water saturation and restoring phase permeability to oil. The introduction of a water-soluble, water repellent into the acid composition minimizes the negative effect on the residual saturation of the pore space caused by the
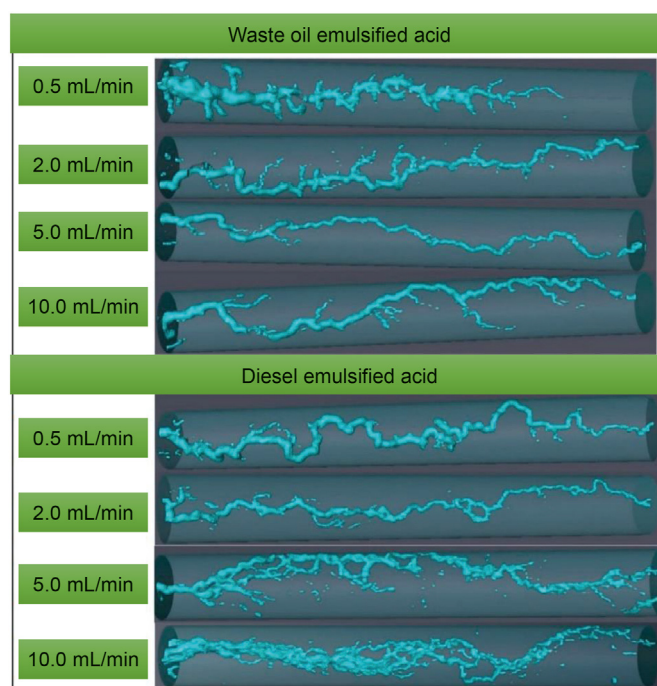

Fig. 10. Wormholes formed by several emulsified acid systems (modified after Ahmed et al., 2018).

spent acid. It favors a more complete wetting of the pore surface with oil when inflow is caused.

The mechanism of action of VES acid compositions is based on their ability to repeatedly increase viscosity during reaction with carbonate rock.

In water-saturated intervals, when the acid composition reaches the formation and reacts with the carbonate rock, it loses its concentration. The pH of the solution increases, and the presence of divalent metal ions, particularly Ca^{2+} and/or Mg^{2+}, force the VES molecules to form long rod-like micelles resulting in high viscosities (Fig. 11a). As the viscosity increases, the composition effectively diverts the next dose of fresh acid to the untreated zones to stimulate a further volume of the target formation. This is a continuous diverting process perpetuated during the acidizing treatment (Kelland, 2014). The resulting gel creates an effective, localized deflection of new portions of the acid solution to previously untreated, low-permeability areas of the formation, with all the VES acid eventually breaking down after the treatment.

In the presence of hydrocarbons in the target zone, viscoelastic properties are not effective and the viscosity of the solution remains approximately unaltered from the viscosity of the original solution. This preserves the reservoir properties of the zone and increases the efficiency of well development after the bottomhole treatment (Xie et al., 2022b). The main reason for the rheological change in the reservoir fluids after VES acid treatment in hydrocarbon-bearing zones is that elongated micelles obtain a spherical form (Fig. 11b) causing fluid viscosity to be dramatically reduced and homogenized (Lynn and Nasr-El-Din, 2001; Sokhanvarian et al., 2017; Yan et al., 2019).

VES-based gelled acids can be effectively used in a variety of reservoirs and wells: porous and fractured-pore carbonates with significant layer-by-layer heterogeneity; with large-volume HCl treatments; multilayer reservoirs; reservoirs with thick productive intervals; reservoirs with non-uniform permeability; for horizontal and vertical wells (Abdrazakov et al., 2019). To increase flow in high-water-cut, heavy oil wells, VES-based gelled acid treatments can also be utilized. By chemically redirecting flow, the treatment temporarily blocks flow from water-bearing zones while efficiently stimulating oil-bearing zones. Moreover, the VES-based gelled acid system has the unique, specific property of limiting acid penetration into highly water-saturated zones, thereby diverting it to penetrate deeper into highly oil-saturated layers (Al-Sadat et al., 2014). The first application of VES acid systems occurred in 2001 (Kelland, 2014). The viscosity of the VES system was shown to depend on pH and it demonstrated excellent diversion capability due to its stable viscosity. Despite many tests performed on VES-based gelled acid systems, specific data on the optimal ratios and total concentrations remains unavailable. Most likely, this is because the VES-based self-diverting acid (SDA) system is a relatively new class of acidic treatments. Rheological characteristics of VES-based SDA have been studied experimentally, e.g. for the Novel-Sulfobetaine VES-based acid system (Cetin and Nasr-El-Din, 2017). The investigated composition exhibited a viscoelastic behavior over a substantial temperature (78–350 °F) and shear rate (10–935 s^{-1}) ranges. By studying the impact of additives including: corrosion inhibitors, H2S scavengers, and iron control agents, the rheological behavior of spent acid was shown to depend on the surfactant and salt type and concentration, temperature, and acid additives (Cetin and Nasr-El-Din, 2017).

Wu et al. (2019) investigated the performance of a VES-based acid solution on the uninvaded matrix. The flooding experiments were conducted using real cores to analyze the impact of VES/salt (CaCl$_2$) concentration on solution rheological behavior. Scans of the cores after the flooding tests revealed a comprehensive description of their inner structure. The findings confirmed that a large pressure difference played a role in fracture propagation, and that the relative permeability of water rose dramatically after the tests (Wu et al., 2019).

Chieng et al. (2020) investigated the performance of modified VES to thicken fracture stimulation fluid. These fluids were created by combining a long-chain cationic surfactant (cetyltrimethylammonium bromide - CTAB) with organic acids (citric acid and maleic acid) in 3: 1 and 2: 1 M ratios, respectively. CTAB and citric acid (CA) were coupled to create a CTAB- based VES fluid with highly effective fracturing properties (Chieng et al., 2020).

Molchanov et al. (2021) experimented with long-lived wormlike micelles containing zwitterionic and anionic surfactants combined to produce a temporary stable network. A combination of oleylamidopropyl dimethyl betaine and sodium dodecyl sulfate exhibited highly stable wormlike micelles at a relatively low concentration (12–32 mM). These properties made that surfactant combination suitable for generating elastic surfactant gels with small concentrations of readily accessible surfactants (Molchanov et al., 2021).

Furthermore, effective assistance in optimizing reservoir stimulation acid fracturing technology, can be gained by employing a large-scale, true triaxial acid fracturing model to perform tests related to deployments in highly deviated wells (Sahu et al., 2019; Zhang et al., 2022). Fatigue acid fracturing in limestone was evaluated for four samples by applying this technique to highly deviated well conditions to investigate the interaction of acid fractures, calcite veins, and weathering zones (Zhang et al., 2022). Fig. 12 summarizes the results of these tests. In sample #1 the acid fracture began adjacent to the wellbore, and an asymmetrical and longitudinal double-wing fracture formed. In sample #2 the acid fractures emerge from the bottom of the well and propagate outwards from that point. The single-wing fractures grow synchronously following natural fracture trajectories. Fracture propagation along other trajectories is inhibited by the influence of the natural fractures present, indicating that they act as barriers. In sample #3, at the first cluster, the injected acid preferentially followed natural fractures rather than penetrate the rock matrix. At points where two fractures intersected, the natural fracture became the primary fracture, along which the acid propagated until it broke through the surface of the test rock sample. In sample #4, three clusters were successfully activated using a fatigue acid fracturing strategy, and a fracture with a more complex morphology was formed emanating from a horizontal well trajectory.

The effectiveness of self-diverting acids tested in such triaxial model experiments depends on their pH. For instance, steering agents based on Surfogel at concentrations 5%, showed that as its pH increased the hydrocarbons in the formation mobilized by the steering agent increased in viscosity (Bulgakova et al., 2013; Zhang et al., 2022).

The advantage of modern VES-based, gelled acid treatment is that it leaves little or no residue (formation damage). However, in its current form, it is more expensive to deploy compared with polymer-thickened acid treatment, although the use of these compositions has shown the greatest technological efficiency to date (Liang, 2022; Othman et al., 2022).

The advantages of gelled-acid compositions for stimulating carbonate reservoirs are (Kelland, 2014):

- The ability to regulate the final viscosity of the self-diverting acid composition by changing the concentration of the gelling agent over a wide range, depending on the objective (Chacon and Pournik, 2022);
- Increased efficiency of acidizing due to fluid loss reduction in cavernous formations;
- Absence of colmatation products in the pore space due to the complete destruction of the gel after treatment;

Table 4
Most-commonly used emulsified acids compositions for carbonate reservoir stimulation.

Type of oil	Type of emulsifier	Type of acid	Inhibitors/ particulate	Ref.
Diesel	Cationic surfactant emulsifier	15% HCl	Used/-	Sayed and Nasr-El-Din (2013)
Diesel	Surfactant	20% HCl	Used/particulate agents (clay), anti-sludge, iron control, emulsifier	Al Mubarak et al. (2015)
Waste oil	Cationic surfactant emulsifier	15% HCl	Used/-	Sidaoui et al. (2017)
Diesel	15 of hydrophilic surfactant (HLB), 4.3 of lipophilic surfactant (LB)	15% HCl	-/-	Taiwei et al. (2018)
Waste oil Diesel	Cationic surfactant emulsifier	28% HCl 28% HCl	Used/particulate agents (nano clay) to achieve significant thermal stability	Ahmed et al. (2018)
Kerosene	Alkonat L90 (nonionic surfactant) + n-butanol	1.5%, 5%, 10%, and 15% HCl	Used/-	Neuma et al. (2020)
Diesel	Cationic surfactant emulsifier	15% HCl	Cationic ammonium/-	Cairns et al. (2016)

Table 5
Experimental specifications and conditions used to test emulsified acids applied to carbonate core samples.

Type of oil	Type of core samples	Type of emulsifier	Rate of injection, cm³/min	Pressure, MPa	Temperature, °C	Ref.
Diesel	Homogenous Indian limestone. Diameter 1.5 in, length 12 in, permeability 3–6.5 mD.	Cationic emulsifier	5	48.2633	150	Cairns et al. (2016)
Waste oil Diesel	Indian limestone. Diameter 1.5 in, length 12 in, permeability 2–4 mD, porosity 15%.	Cationic emulsifier	5 2	20.68427	135	Ahmed et al. (2018)
Kerosene	Jandaíra formation. Diameter, 1.338 in, length 1.9685 in.	Alkonat L90 (nonionic surfactant) + n-butanol	1	13.78951	30	Neuma et al. (2020)
Diesel	LF formation (Jurassic limestone reservoir). Permeability 1–15 mD, porosity 5%–20%, H₂S 7%.	Surfactant	2-4	68.94757	115.556	Al Mubarak et al. (2015)
Diesel	Dolomite. Permeability 1.6-20.9 mD, porosity 9.3%-14.02%, H₂S 7%.	Cationic surfactant emulsifier	0.5-10	13.78951	148.889	Sayed and Nasr-El-Din (2013)

Table 6
Experimental test results of emulsified acid treatments applied to carbonate reservoirs.

Type of oil	Type of emulsifier	Optimal concentration	Findings	Ref.
Diesel	Cationic and nonionic emulsifiers	15% HCl, 70:30 acid-oil-ratio, 2 wt%, HLB (hydrophile-lipophile balance) value 5.	Fluid-stable W/O emulsions were obtained without phase separation at room temperature and high temperature (90 °C). Using two emulsifiers in combination gave better results than using cationic or non-ionic emulsifiers alone.	Song and Lee (2021)
Diesel	GF–15 MPS Surfactant (mixture of alkyldimethylbenzonium chloride, tertiary amine, stabilizer, and glycolylic solvent)	15% HCl, 0.5% GF–15 MPS Surfactant	When applied, upper zone permeability rose from 110 to 1632 mD, and active batch diversion increased from 4.6 to 470 mD. The enhanced permeability in both layers justified the efficacy of the intended stimulation strategy. The viscosity and thixotropy tests revealed that the generated emulsion used as a diverter was easy to flow back and generated the least amount of post-treatment residual damage. In contrast to other mixtures tested, by increasing the neutralization time, combining 15% HCl and a surfactant with a concentration of 0.5 % yielded the best reaction rate and dissolution rate. The majority of the stimulation fluid is a mixture of HCl and surfactant at concentrations of 15% and 0.5 %, respectively.	Jafarpour et al. (2019)

- The ability to form a branched network of wormholes in the target zone, not only because of the heterogeneity of the rock, but also because of the redirection of the impact vector on the rock as a result of the increased resistance of the formed gel;
- Does not require high rates of fluid injection; and,
- No requirement for expensive specialized equipment to implement the treatment (Liu et al., 2021; Othman et al., 2022; Rabie et al., 2012).

However, VES-based gelled acid treatments are ineffective for reservoirs with high water cuts. In such conditions, there is a risk of forming gel-like sediments of iron hydroxide as the acids become neutralized and the solution's pH increases during the treatment of a predominantly water-bearing formation.

3.4.2. In-situ polymer-based gelled acids

Polymer gels have a higher viscosity than traditional acid treatment solutions. They will, therefore, tend to penetrate initially into the low-permeable intervals. A viscous solution, such as a polymer gel, can be used in two ways. 1) As a viscous polymer gel tablet which can be injected (or preflushed). The viscosity of the preflush is used to change the injection pressure of the interval it enters. This preflush acts to restrict the injection of additional fluids into that zone when it enters the formation. Other areas of the interval will break down and begin absorbing fluid when the injection pressure increases inside this region of the interval. The damaged zones will then be penetrated by a low-viscosity acid main flush. 2) The acid solution can be gelled. As already explained, the advantage of acidizing with gelled-acid diverters is to slow the
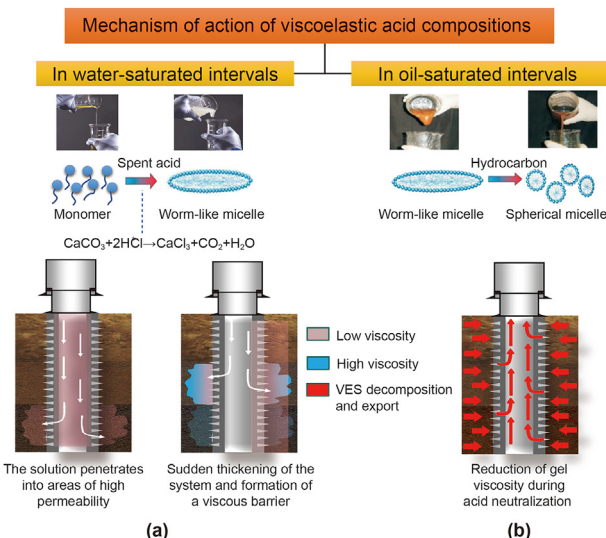
Fig. 11. Mechanisms involved in visco-elastic surfactant (VES) acid treatments of carbonate reservoirs.

acid-rock reaction rates, thereby providing better penetration of low-permeability zones. The gelled solutions also reduced fluid loss due to increased flow resistance because of the high viscosities involved (M. Zhao et al., 2020).

Several mechanisms are associated with polymer-based self-diverting acid systems (Gomaa, 2010; Zhou et al., 2009). When the acid is pumped and reacts with the formation, the pH increases. At a pH value of about 2, the carboxylate groups of the polymer are deprotonated and react with the crosslinking agent to form a gel. This gel has the ability to fill and close formation zones with high permeability and direct any subsequent acid injected to stimulate less permeable or damaged areas. The acid in this formulation is used as a diverting agent. The gel structure is then destroyed when the destructor is activated at a higher pH. The permeability of the plugged area is restored by the backflow of the well. Gel breakers usually reduce the viscosity of the viscous solution after the treatment. The common breakers include sodium erythorbate, sodium persulfate, and destructors made from metal salts, which are ionic materials that primarily form complexes with metal ions, such as fluorides (Fogang et al., 2020; Gomaa and Nasr-El-Din, 2010). Ideally, a breaker should leave minimal residue as the viscosity reduction proceeds. The procedures used to evaluate the suitability of a breaker involve rheology and core-flooding tests.

The most typical polymer-based diverting agents are hydroxyethylcellulose (HEC), acrylamides, and polysaccharides such as guar, xanthan, and succinoglycan. These are readily available and efficient, but few work effectively if the well temperatures exceed 90 °C To overcome this limitation, cross-linked agents and other synthetic polymers such as, N,N-demethylacrylamide, acrylamido propanesulfonic acid, and quaternary monomers have been used (Poelker et al., 2005).

The basic acid solution can be injected with the polymer and a second reagent that crosslinks the polymer under reservoir conditions at elevated temperatures in the well, further increasing the viscosity of the solution. To crosslink polysaccharides, such as guar, acid fracturing often employs tetravalent and trivalent metal salts, including zirconium (IV) and titanium (IV). Crosslinking agents suitable for anionic polymers, such as partially hydrolyzed acrylamide polymers, metal salts such as aluminum salts, iron (III) salts, phenolic compounds and/or low molecular weight monoaldehydes and polyamines are used. Some of these materials are similar in

composition to reagents used for aquifer isolation (Rae et al., 2006). A crosslinking system with aluminum requires no additional reducing or liquefying agent because the crosslinked gel completely reduces viscosity as the acid is neutralized (Daifeng et al., 2019; Patil et al., 2012).

The problem of using thickened or highly viscous polymer solutions is the difficulty of removing its residue that tends to clog the formation. Such clogging acts to inhibit oil flow and production from the formation. It often requires costly cleanup operations. It is therefore prudent to minimize solution thickening associated with polymer-based treatments.

High viscosity fracturing fluids induce high net formation pressure, and are associated with less fracture fluid leakage, but is inhibited from entering smaller micro-fractures. Simple transverse fractures tend to be produced, which can restrict the development of complete fracture networks. By reacting with natural fractures, subsequent injection of low viscosity fracturing fluids can assist in promoting the formation of more complex fracture networks. Consequently, it is often appropriate to initially inject a high viscosity polymer, such as guar gum, followed by the injection of low viscosity treatment fluids to further fracture tight formations along the trajectories of their natural fractures. The use of viscous acid compositions tends to increase efficiency in the use of hydrochloric acid due to the lack of its consumption in cavernous carbonate formations, and the uniform distribution of acidic fluids extending through the formation. Nevertheless, the involvement of relatively expensive, specialized equipment may be required to overcome the resistance forces arising from high viscosities, which can create formation damage that degrade the beneficial effects of the acid treatment. Typically, wells previously exposed to acid treatments and fracture stimulation show a greater increase in productivity when subsequently treated with high-viscosity acids. This is to be expected as more acid is able to penetrate further into the formation at prevailing fracture pressures in such conditions.

Table 7 describes some studies of *in-situ*, polymer-based, and gelled acids.

3.5. Acid treatments with fiber additives

Fiber-based, diverting acid systems are designed to regulate the direction of filtration flows during acid treatments in fractured carbonate reservoirs. The process of oil production enhancement in carbonate reservoirs is complicated by the presence of highly permeable interlayers and fractures in the reservoir zones. Only the highly porous interlayers are exposed to acid treatment at low injection pressures, without effectively penetrating the lower porosity/permeability zones of the formation (Chen et al., 2021; Zhao L. et al., 2020). The fiber-based injected solutions generate temporary blockages in certain reservoir zones, creating a flow-deflecting screen that redirects the acidic compound, in particular, into less permeable zones (Quevedo et al., 2012). The most commonly used fibers for oil/gas reservoir stimulations are polyester fiber, polypropylene fiber, carbon fiber, polylactic acid fiber, and bamboo fiber (Fig. 13) (Pu et al., 2021).

Fibers display a proclivity to flocculate and bridge when interacting with suspended fluids and formation irregularities such as fractures, micro-fractures, and wormholes in carbonate formations. Fig. 14a illustrates the aggregation of fibers. Such an aggregation tends to flocculate into clumps when dispersed as a highly concentrated fluid suspension of fibers, and that suspension is sheared (Fig. 14b). Such flocculation leads to a complex network of intertwined fibers (Fig. 14c). When interacting with fluid flow, fibers' ability to entangle, flocculate, and form a network structure offers beneficial properties for acid stimulation treatments. Fibers offer a relatively low-cost option to promote acid diversion due to
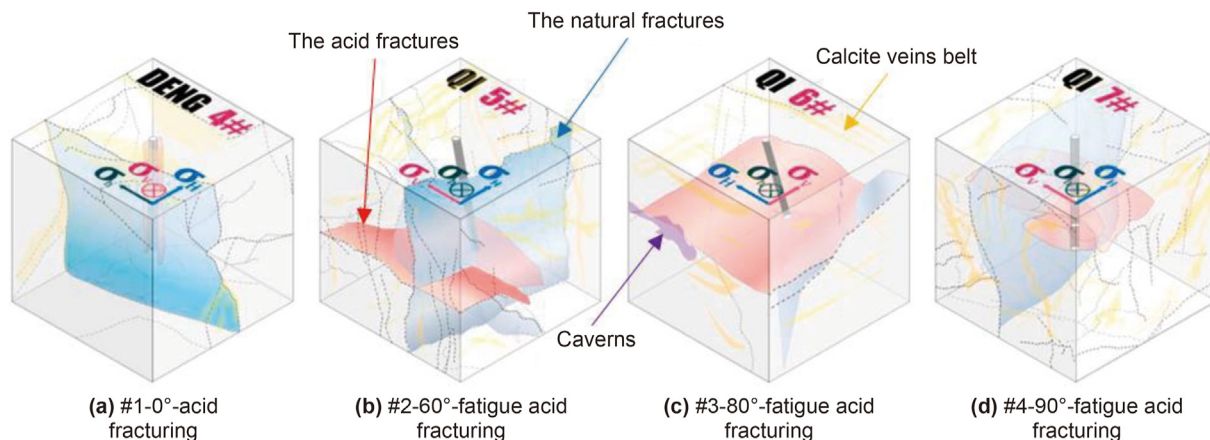
Fig. 12. Fracture morphologies generated by large-scale, true triaxial acid fracturing model experiments (reproduced from Zhang et al., 2022).

this feature (Ghommem et al., 2020; Kraemer et al., 2015).

Fig. 15 illustrates the effects of combining chemical treatments (e.g. acidizing) with a mechanical diverter made of fibers. The fibers generate a net blocking the more permeable zones, thereby enhancing the diversion of the acidizing solutions away from the high permeability intervals (Quevedo et al., 2012).

Mono-sized synthetic fibers are commonly used in hydraulic fracturing (e.g. FiberFRAC (Pooniwala et al., 2022)), to boost the fracturing fluid's proppant carrying capacity and to prevent proppant-laden pulses from dispersing. Guo et al. (2015) used mono-sized synthetic fibers to enhance channel fracturing with hydroxypropyl guar (HPG) at a concentration of 0.50 wt% base gels in fresh water. Their results showed that fibers in HPG base gels form a quasi-three-dimensional network structure, which enhanced the apparent viscosity of the fracture fluid. This suggests that less diversion agent is needed to achieve the same viscosity by involving fibers. An optimum concentration of HPG diversion agent has yet to be determined (Shah et al., 2020). The degradation of fibers at high temperatures causes changes to the rheological behavior of fracturing fluid. According to rheological experiments, the apparent viscosity of a crosslinked-fracturing fluid increased by 12–15 mPa with a 1% fiber content (Guo et al., 2012, 2015). Fig. 16 illustrates that the temperature effect on the apparent viscosity of suspensions is comparable for a range of tested fiber concentrations. For base gels, as the test temperature increased, the apparent viscosity of fiber suspensions decreased (Guo et al., 2015).

Before being injected into a reservoir, fibers are typically softened in a carrier fluid. A filter network (often referred to as "filter cake", not to be confused with the layer of that name drilling fluids form on borehole walls) is a three-dimensional network structure formed by long and short fibers wrapping around one another (Wang et al., 2015; Yang et al., 2019). Fig. 17 illustrates the three processes involved in the creation of an effective fiber-based plugging layer.

(1) *Inner filter cake fabrication stage.* Following the pumping of fibers into the reservoir, they preferentially flow towards the high permeability layers and fractures, following pathways of least resistance. The longer fibers tend to become entangled as bridges at pore throats or fractures. The displacement pressure forces the fibers into the pore throats or inner fractures. The drag resistance and different degrees of pore wall roughness act to hold the fibers in place (Fig. 17a).

(2) *Exterior filter cake fabrication stage.* The trapped long fibers act as bridges and their accumulation increases as the fluid injection process progresses. At the same time, the ability of the coarse pore/fracture walls to capture short fibers is improved. The fibers become entangled with each other, and a layer of fibers is gradually built up during this capture process (Fig. 17b).

(3) Fiber layer compaction stage. The fibrous layer is continuously compressed due to the pressure difference between the outer and inner parts (Fig. 17c). The filtering rate is shown by $v_{1,2,3}$ in Fig. 17. The filtering rate v_3 is almost equal to 0 once a thick filter cake is established. Once stimulated, these fibers dissolve in acid, alkaline solution, or water and flowback into the wellbore during subsequent production (Yang et al., 2019; Zhao L. et al., 2020).

Fibers can also be used with other composite diverting agents including chemical particles, gels, and VES. The benefits of each diverting agent class are combined in composite diverting agents. Such combinations may be able to satisfy the reconstruction demands of various reservoirs. Fibers and chemical particles are two common composite diverting agents utilized in oil-field stimulation treatments. The chemical components typically involve KCl and $NaNO_3$ (Al-Othman et al., 2017; Che et al., 2018; L. Zhao et al., 2020; Zhu et al., 2020) or self-diverting acids (Abdollahi et al., 2021; Jauregui et al., 2011), and sometimes both (Shi et al., 2015; Zhang et al., 2018). Fig. 18 illustrates how diverting agents can help to re-orient new fractures emanating from existing fractures. Composite agents consisting of fibers and chemical particles act to temporarily block existing fractures, allowing new fractures to form and spread, improving communication between fractures and the wellbore in the near-well zone, and enhancing well productivity (Zhao L. et al., 2020).

Variations in differential pressure and the flow of fiber suspensions during fiber injection influence the magnitude and direction of the newly-formed diverted fractures. A numerical model developed in conjunction with laboratory trials provides insight with which to assess leak-off in bridges formed in porous media with fibers (Ghommem et al., 2020). Fig. 19a plots the transient fluctuations in differential pressure (between inlet and outflow) obtained by the computational model. The trend depicts fiber flocculation and flow towards the pipe exit. The differential pressure is relatively initially but rises as the fibers entangle and form a blockage at the pipe's exit. The pressure rises as the fibers aggregate

and obstruct the passage of fluid through the exit constriction, eventually reaching a peak. The pressure increase is a representation of the bridge's resistance. When the fibers escape the pipe completely, the pressure drops dramatically and stabilizes at a low value. The computational results agree with the experimental measurements of the pressure trend following fiber injection (Fig. 19b). It just takes a few seconds for flocculation and bridging to occur (Fig. 19c). The trials were carried out utilizing laboratory-scale equipment designed to investigate leak-off behavior. The simple experimental equipment (Fig. 19d) consists of a tube with a piston, a pump, and an opening. The composition of fibers and acid are forced through the piston, and the transient pressure is measured when the fiber-laden acid forms a plug and passes through the constricted aperture (Ghommem et al., 2020).

3.5.1. Mechanism associated with fibers deployed with acid treatments

Fractured carbonate reservoirs are the most difficult to control from the perspective of acid stimulation treatments. This is because such reservoirs display extremely strong permeability contrasts. The fractured sections of such reservoirs can have permeability several orders of magnitude higher than undisturbed matrix sections. Even when injecting solutions with deflecting additives, such as visco-elastic polymer-free acid (VDA), the greatest challenge is to block fractures and treat the rest of the reservoir. Therefore, large volumes of the solution have to be pumped into the well, often increasing the cost of the stimulation with modest results.

Schlumberger launched the MaxCO$_3$ system in 2007 to investigate novel fibers as additives to deflect fluids in open-hole and cased-hole wellbores. That system created blockages with fibers and enhanced viscosity by consuming acid, resulting in an intermediate block and reducing the rate of fluid seepage into natural fractures and wormholes (Fig. 20) (Bukovac et al., 2012;

Schlumberger, 2014).

Engineers found that simply adding fibers to a normal HCl solution does not create a stable suspension of fibers in a basic acid solution. After adding the fibers, clumps tended to form and drop out of the solution. The success was achieved due to the combination used in the MaxCO$_3$ system of degradable fibers and VDA. That combination made it possible to create a stable suspension of separate fibers in the solution (Bukovac et al., 2012; Schlumberger, 2014). Fig. 21 compares the laboratory test results of solution absorption times for a VDA system and the MaxCO$_3$ system.

During the experiment, the VDA and the MaxCO$_3$ acid systems were pressed through a 2-mm hole and channel simulating a natural fracture. The graphical relationship confirms that after a short time the MaxCO$_3$ acid system was able to form a stable crust on the channel walls. When testing the VDA system, a similar effect was not obtained, and the fluid was absorbed into the formation at a constant rate.

Experiments were carried out in an apparatus with holes of different diameters, through which the acidic liquid with fibers was passed at different speeds. Circular holes with a diameter of 1—2 mm (0.04—0.08 in) were used to simulate the channels created during acid treatment. Rectangular holes 2—6 mm (0.08—0.24 in) wide served as analogs of cracks. During the experiments, fiber plugs formed and the pressures at which acid with the fibers passed through the holes were recorded. The experiment was conducted with a liquid MaxCO$_3$ Acid© with a mass content of 15 % VDA solution and fibers at a concentration of 6 kg/m^3 (Abdollahi et al., 2021).

At first, the pressure did not increase (zero period, when the fibers had not yet formed a plug), but after a few seconds, there was a rapid increase, caused by the formation of a plug by the fibers, which blocked the hole (period #1). The pressure continued to rise until all the acid had reacted. Experimental results showed that

Table 7
Studies of *in-situ*, polymer-based, gelled acids.

| Type of polymer	Optimal concentration	Work conditions	Findings	Ref.
Polyacrylamide emulsified	20 mL/L of polyacrylamide, 5 wt% HCl	Laboratory (121 °C, 5.5—6.2 MPa).	At low shear rates, the *in-situ* gelled acid created a gel inside the core, as indicated by the cyclic behavior of the core pressure drop, and the acid changed orientation multiple times inside the core. On the other hand, this gel significantly reduces the permeability of the core. At high shear rates, less gel is formed inside the core, and the ability of the acid to change direction inside the core is lower than at low shear rates. Consequently, a significant increase in permeability has been achieved. The length of the wormhole increases with the shear rate, but the width of the wormhole increases with the cumulative volume of acid injected.	Gomaa (2010)
Polyacrylamide which consists of dimethylaminoethyl acrylate methyl chloride quarternized (90%), and acrylamide (10%)	2.5 vol% of polyacrylamide, 5 wt% HCl, 1 vol% FeCl$_3$, and 1 vol% corrosion inhibitor	Laboratory (121 °C, 0.48 MPa).	The viscosity and elastic modulus of *in-situ* gelled acid increased with acid expenditure (pH up to 4.5). It revealed a self-breaking ability when pH surpassed 4.5. The inclusion of salts changed the polymer's structure and caused it to split from the acid solution, lowering the viscosity of the *in-situ* gelled acid system. The best flow rate for the smallest volume of PVBT injected was 5 cm^3/min. The acid system was capable of diverting with permeability disparities as high as 4.6, stimulating up to 50% of the low-permeability core at permeability contrasts as high as 8.8. The injected solution was compatible with the crude-oil system.	Sarmah et al. (2020)
Acrylic polymers and oxyalkylated alkyphenols	5 wt% HCl, 20 gal/Mgal of polymer	Laboratory (38 —65 °C, 3.45 MPa).	The polymer formed a gel within a restricted pH range. The viscosity of the acid increased *in-situ* as a result of gel formation, and acid diversion became possible. The gelled acid was able to generate wormholes that are uniformly dispersed throughout the target zone. This gel enhanced acid placement and offered more consistent damage elimination. It also kept acid fluid loss under control. Furthermore, due to the shear-thinning action of the polymeric gelling agents, viscosity is reduced with an increasing shear rate.	Taylor and Nasr-El-Din (2003)

Fig. 13. Images of five different types of fibers used in oil and gas sub-surface stimulations: (**a**) polyester fiber, (**b**) polypropylene fiber, (**c**) carbon fiber, (**d**) bamboo fiber, and (**f**) polylactic acid fiber.
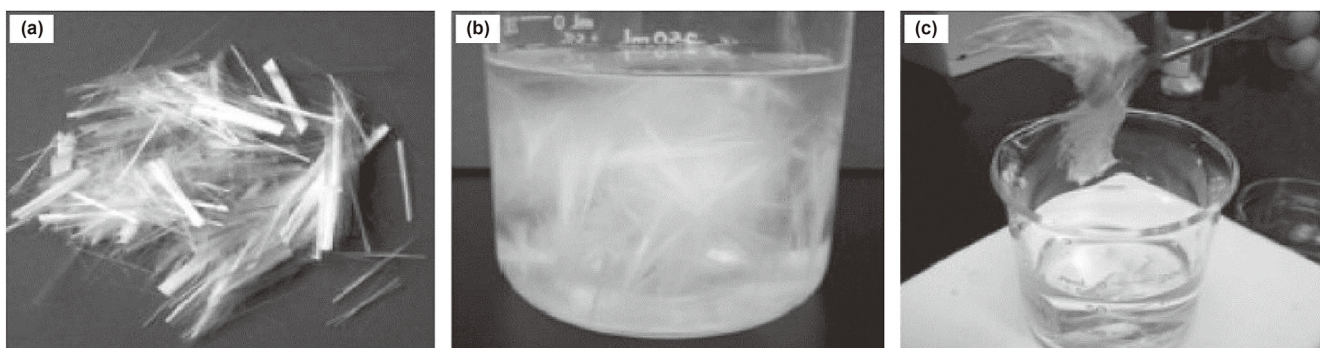
Fig. 14. Flocculation of fiber agglomerations in suspension: (**a**) fibers in dry form; (**b**) fibers dispersed in a water-based solution; (**c**) a network structure made up of intertwined fibers (adapted from Ghommem et al., 2020).
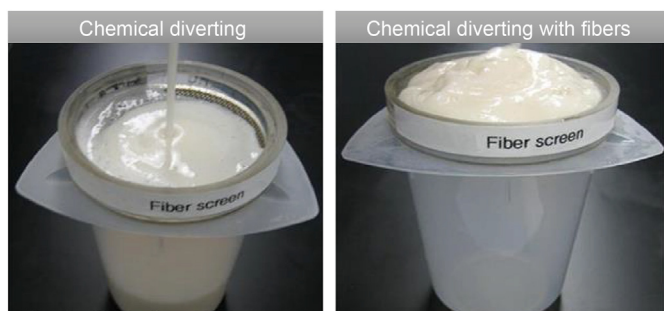
Fig. 15. Illustration of the benefits of combining chemical treatments with mechanical (fiber) diversion at 135 °C to divert the chemical treatment away from the more permeable zones (modified after Quevedo et al., 2012).
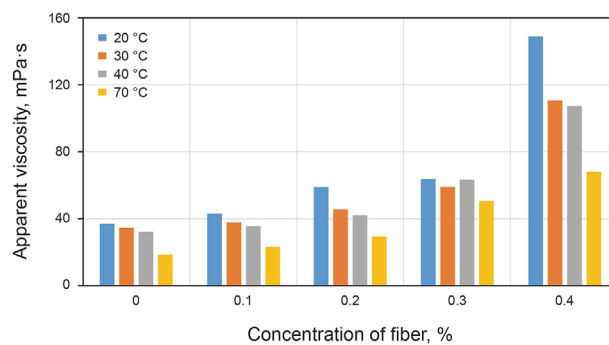
Fig. 16. The influence of temperature on apparent viscosity at a shear rate of 118 s^{-1} (reproduced from Guo et al., 2015).
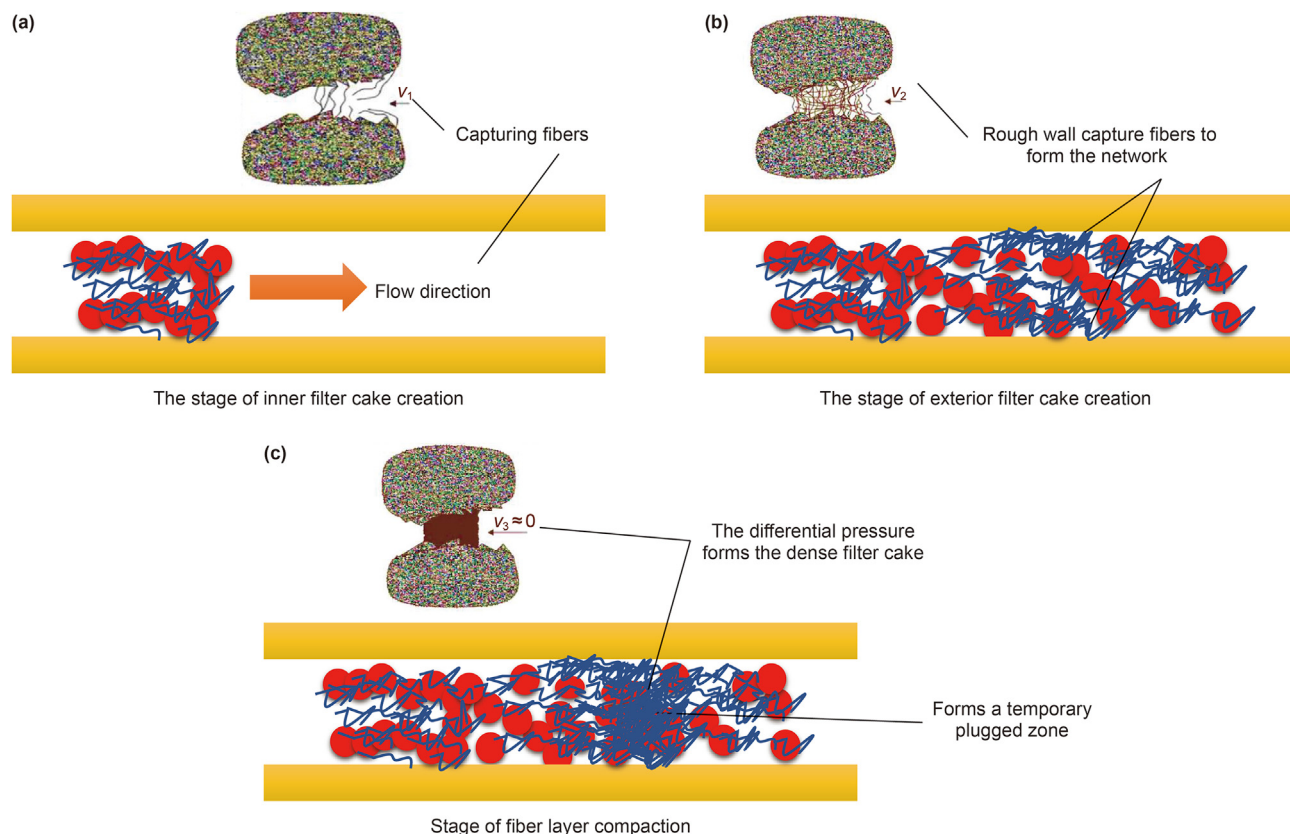
Fig. 17. Diagrammatic representation of a temporary reservoir plugging mechanism involving fibers (modified after Yang et al., 2019).

when acid with fibers entered the perforations, the acid passed into the formation as if it contained no fibers. Then, when a plug was formed, the fibers accumulated in front of the perforations and formed a filtration crust. The fibers then clogged the perforations, reducing the injectivity of the interval and directing the acid to other perforations. It was also found that the concentration of fibers in the acid liquid required to plug the perforation holes increased as the pumping rate increased. After pumping acid with fibers through the perforation, the model was flushed with fresh water (period #2). As the viscous acid left the model, the injection pressure gradually decreased and finally stabilized (period #3). At the end of each experiment, a solid plug of fibers was left in the hole. Knowing the pressure, injection rate, fluid viscosity, and length of the fiber plug, using Darcy's law, the permeability of the plug can be calculated. Depending on fiber concentration and pumping speed during plug formation, the measured permeability varied from 0.4 to 2.4 μm^2. These results imply that fibers are the most effective means of deflection in intervals with a permeability greater than 0.1 μm^2 (Abdollahi et al., 2021; Asiri et al., 2013).

Fibers added to an initially low-viscosity of viscoelastic self-dissolving acid (VSDA) have been shown to react with a carbonate formation to create viscosity and assist in bridging fractures and perforation tunnels (Pooniwala et al., 2022). After treatment, the VSDA and fibers dissolve and completely disappear, leaving no damage to the formation, as illustrated by the sequence displayed in Fig. 22.

Fibers can act to limit acid loss in existing wormholes and high permeability zones by temporarily plugging perforations and redirecting flow to less-permeable perforated zones (Fig. 23) generating new fractures and wormholes. Similar to the use of

proppant in hydraulic fracturing, low-temperature (LT) fiber was initially introduced and then further developed to use high-temperature (HT) fiber for acid fracturing (Al-Othman et al., 2017; Zhang et al., 2020a).

3.5.2. Mechanism associated with fibers deployed in acid fracturing

In acid hydraulic fracturing, the use of fibers reduces acid escape into both natural fractures and those formed by previous hydraulic fracture stimulation treatments. Combined with deflecting surfactants (VES), acid hydraulic fracture stimulation offers several advantages:

- The relatively high viscosity of the acid (compared to HCl) provides increased fracture width, which reduces the area-to-volume ratio and increases the interaction time between the acid and the rock.
- Reduced water yield increases injection time by reducing the rate at which acid migrates along the fracture surfaces.
- VES-deflecting acids thicken in the injected flow column behind the fiber briquette, acting to reduce instantaneous fluid losses and helping to create an effective temporary barrier.
- The combined action of mechanical and chemical diversion increases acid flow efficiency, resulting in a more effective stimulated fracture network.

The fiber used in such systems can be a non-toxic, low-density synthetic fiber that does not affect the chemical properties of the VES system (Abdollahi et al., 2021). The VES then retains its rheological properties after filtration through the fiber plug and after contact with the formation. Laboratory studies have shown that
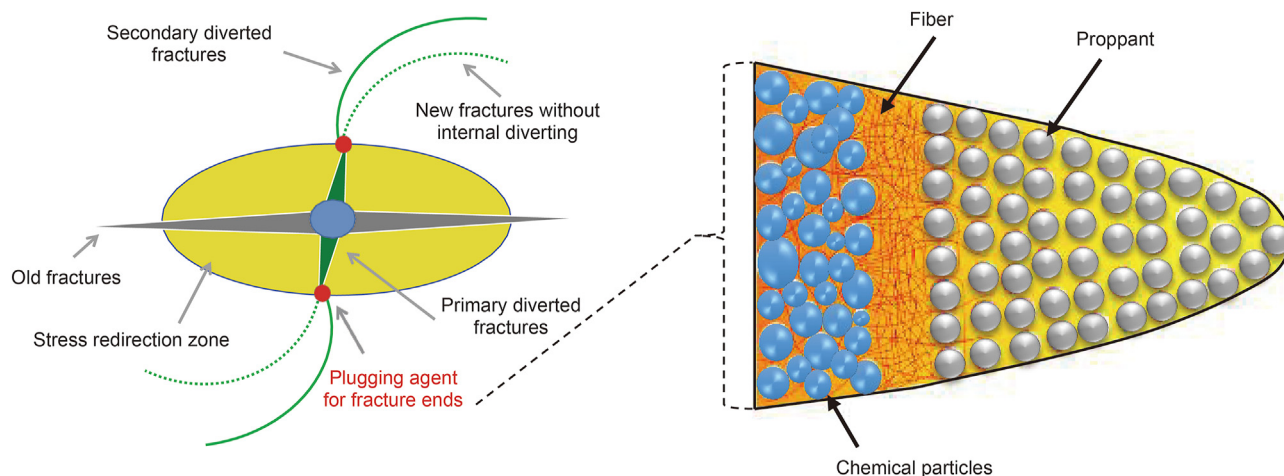
Fig. 18. Diverting mechanism of fibers and chemical particle composites in fracture stimulation (modified after L. Zhao et al., 2020).
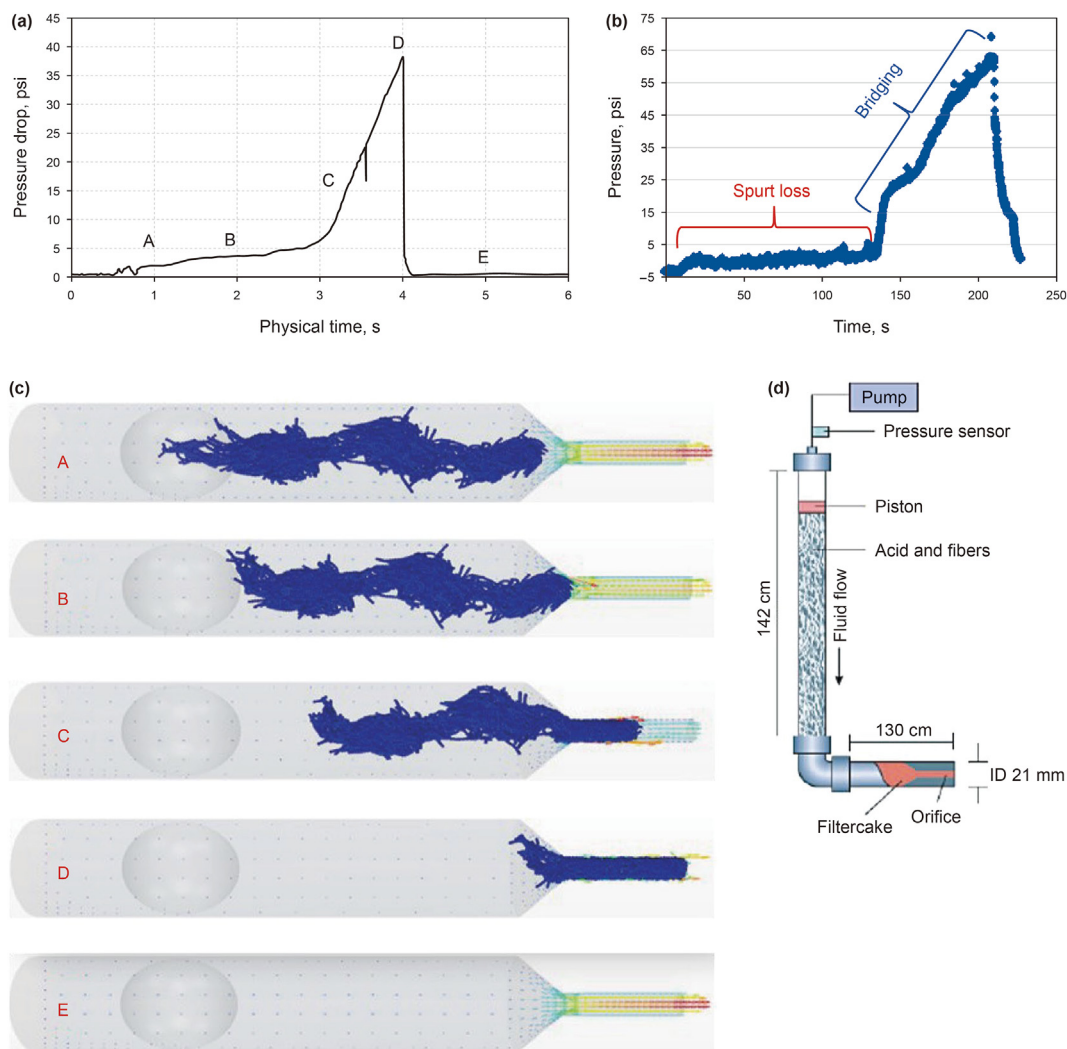

Fig. 19. (**a**) Time versus pressure differential calculated in a numerical simulation. (**b**) Pressure versus time signal recorded during an acid bridging experiment using fiber. (**c**) Several snapshots (A to E) of a fiber suspension flow during the experiment. The fibers are first arranged at random. As they get closer to the "pore throat" representing the pipe exit, they flip and reorganize. (**d**) Experiment setup and fiber plug samples made in flow channels of different shapes (Adapted from Ghommem et al., 2020).

such fiber networks are capable of creating permeability barriers several orders of magnitude smaller than 100 mesh sand. However, unlike sand or many other deflecting solids, the fibers decompose when subsequently treated with an organic acid, making it easy to remove them from the formation after acid treatment. Thus, such systems avoid generating an insoluble residue with the potential to damage the treated formation by reducing its permeability (Abdollahi et al., 2021).

The plugging agent injected into the wellbore can be controlled to seal just some of the perforations connected to some of the open fractures present in the formation, creating more fractures from other perforations as part of multi-stage stimulation (Fig. 24a). Similarly in lateral directions within the formation, some open fractures are temporarily blocked by the plugging agents, causing more fracture branches to develop and progressively expanding the volume of the stimulated reservoir (Fig. 24b). After stimulation, the plugging material is completely destroyed by an organic acid wash, restoring the original conductivity of all fractures. Using fracture networking technology, deep and ultra-deep oil and gas reservoirs can be safely, economically, and effectively stimulated in this way (Xiong et al., 2018). Fibers offer an excellent temporary plugging mechanism for small fracture widths. On the other hand, fibers are less effective at plugging larger fracture widths. As a result, fibers are frequently employed in conjunction with other diverting agents to increase plugging strength. Furthermore, when deploying fibers, displacement needs to be sufficiently decreased to allow the fibers to easily accumulate and snag in the fractures.

Plant fibers and their modified derivatives are mostly used in acid reservoir stimulation treatments (Evgenievich Folomeev et al., 2021; Zhou et al., 2022). The most common types of fibers used are polylactic acid fiber, polyester fiber, and polypropylene fiber (Evgenievich Folomeev et al., 2021; L. Zhao et al., 2020; Zhou et al., 2022). However, fiber properties and capabilities need to be taken into account. Fibers tend to be unaffected by the salinity of the formation fluids encountered, and they can be used across a wide temperature range of about 20—135 °C. As the fibers are manufactured above ground, their properties tend to remain stable when injected into subsurface formation. This is not the case with
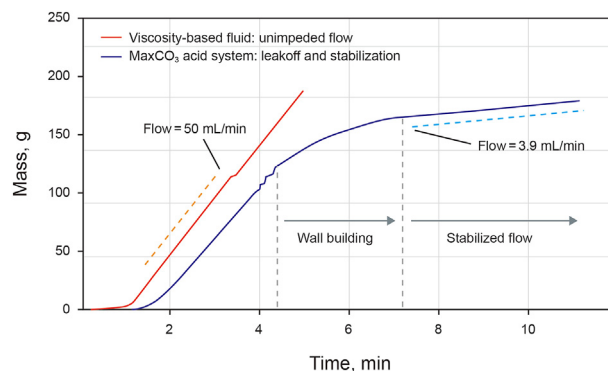

Fig. 21. Time dependence of solution absorption at 1.4 MPa constant pressure for a basic VDA and the MaxCO$_3$ (degradable fibers plus VDA) systems (reproduced from Schlumberger, 2014).

polymers that acquire their crosslinked shapes after injection, as they are impacted by a variety of conditions in the reservoir. In comparison with large-sized particles (e.g. most polymers), fiber insertion into the formations' fractures and pore spaces is comparatively simple. Fibers have soft textures and shapes that can be readily suspended in a range of carrier liquids, with which they typically combine. In practice, fiber injection and its ability to pass through a well completion depend on fiber length, concentration (typically $\leq 2\%$), and chemical composition (Abdollahi et al., 2021; Crowe C et al., 1992; Goenka et al., 2014). Specific fibers typically have a single solvent medium that can be used to ultimately dissolve them. Typically, that solvent is an organic acidic solution. The plugs are therefore readily removed with an organic acid wash after the fracture stimulation operation is completed (Abdollahi et al., 2021; Jiang et al., 2015; Pu et al., 2021; Qi et al., 2013). Fiber performances can be improved by deploying them in conjunction with other diverting agents, since in isolation their plugging strengths are limited. The fiber filter cake layer is denser than chemical particles, resulting in reduced fluid filtering loss, coupled with the ability to effectively prevent proppant reflux. As a result,
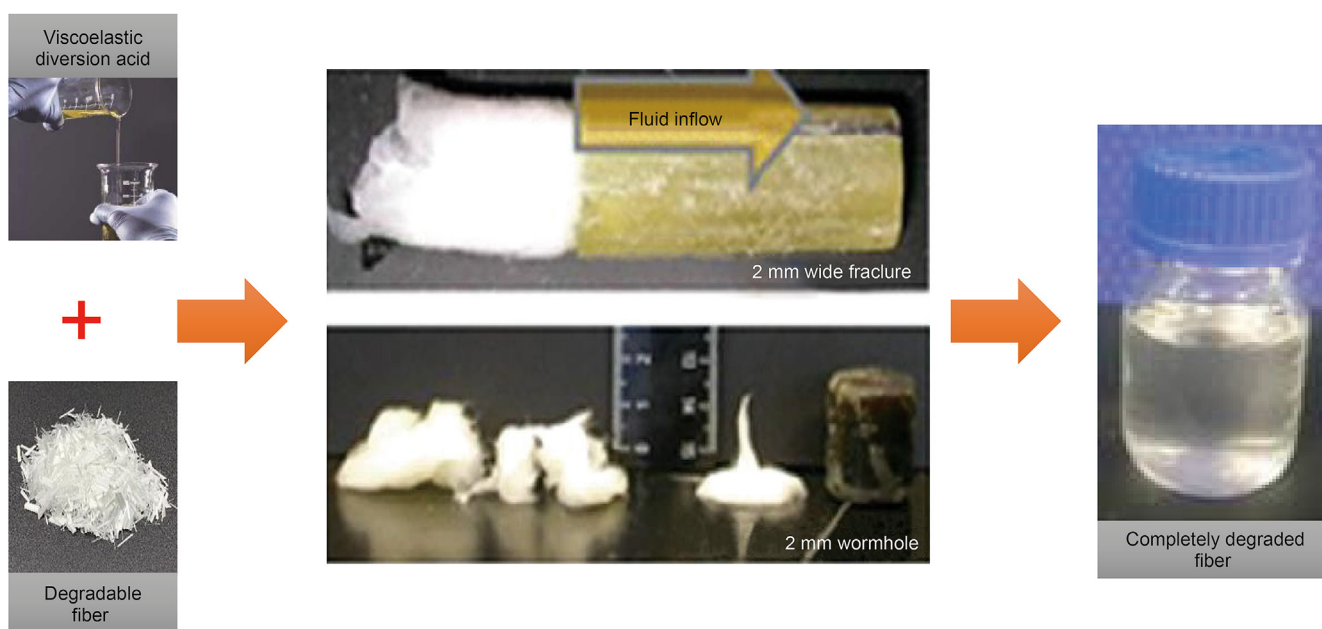

Fig. 20. MaxCO$_3$ system, mechanism of fiber deflection and breakage after processing (photos from Bukovac et al., 2012; Schlumberger, 2014).

fibers can be designed for use with a range of other diverting agents selected to suit fracturing objectives and formation properties (Che et al., 2018; Qi et al., 2013; Zhao L. et al., 2020; Zhou et al., 2022).

The main advantage of fibers is that they can be evenly disseminated in the injecting medium. This property means that fiber-based injection fluids have a high pumping capacity and pose a reduced danger of wellbore obstruction during the injection. Fibers can also interact with proppants to form a solid composite mesh structure, successfully preventing proppant backflow and improving the sand-holding capacity of the fracturing fluid. However, if the injectable fiber concentration is too high, "fiber clumps" may form due to insufficient entanglement dispersion or rapid surface hydration, which will render the injected fluid ineffective. As fibers are highly resistant to most acids, they can be used to form a range of acid-resistant structures (Jauregui et al., 2011; Yang et al., 2020; Zhang et al., 2020b).

On the downside, the current range of available fibers displays poor temperature endurance above 135 °C. Also, the soft textures of fibers make them less effective in unconventional reservoirs with low porosity and low permeability, or in reservoirs with large/cavernous pore space and/or smooth fracture surfaces (Yang et al., 2020).

The constraining criteria for using fibers as a temporary plugging agent with acid stimulation treatments under reservoir conditions are (Chen et al., 2021; Quevedo et al., 2012; Zhao L. et al., 2020):

- Stable over a temperature range of 20–135 °C;
- In most cases, fiber concentration in injected fluids should be less than about 2%;
- Fiber plugging strength is relatively low, ranging from 0.4 to 3.5 MPa;
- Plugging strength diminishes rapidly as reservoir temperature rises;
- Most fibers are soluble in certain organic acids, with an acid solubility rate of 96%; and,
- Fibers are unaffected by reservoir pressure.

Tables 8 and 9 describe the performances of fiber-acid diverting stimulation conditions in laboratory tests and field applications, respectively.
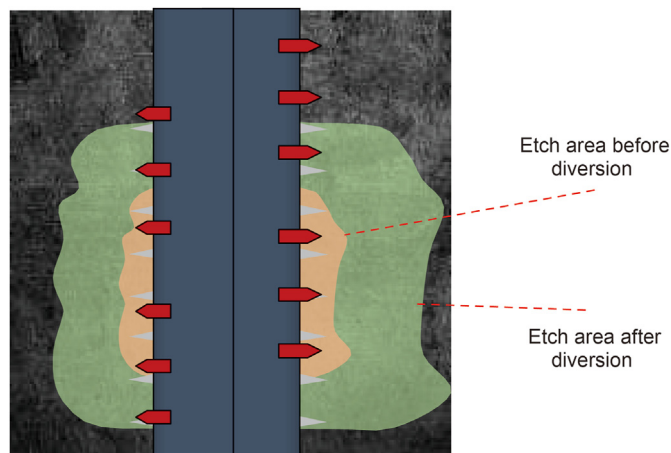

Fig. 23. Schematic representation of injected fluid diversion using an FLVSDA system during acid fracturing (modified after Pooniwala et al., 2022).

4. Nano-acid stimulation methods applied to carbonate reservoirs

Applications of nanotechnology to generally enhance oil recovery is receiving much research attention with promising results (Al-Shargabi et al., 2022a, 2022b; Davoodi et al., 2022). Nano-acid treatment methods applied to carbonate reservoirs are also showing encouraging results (Kalgaonkar et al., 2022; Kim et al., 2020). A nanofluid is a fluid in which NPs are well distributed and do not collide. The stability and distribution of NP in nanofluids is therefore an important consideration. McElfresh et al. (2012) proposed that by altering their charge density and zeta potential, NP can remain stable under a wide range of reservoir conditions. Furthermore, Zhang et al. (2014) discovered that NPs are able in certain conditions to minimize residual oil saturation by detaching oil molecules from the rock surface, resulting in a change in wettability. The two fundamental constraints affecting NP stability in porous environments are particle aggregation and pore throat occlusion (Al-Shargabi et al., 2022a; McElfresh et al., 2012; Zhang et al., 2014).

Kim et al. (2020) studied the effect of nano-acid fluids on improving oil recovery in tight carbonate reservoirs. SiO$_2$-acid,
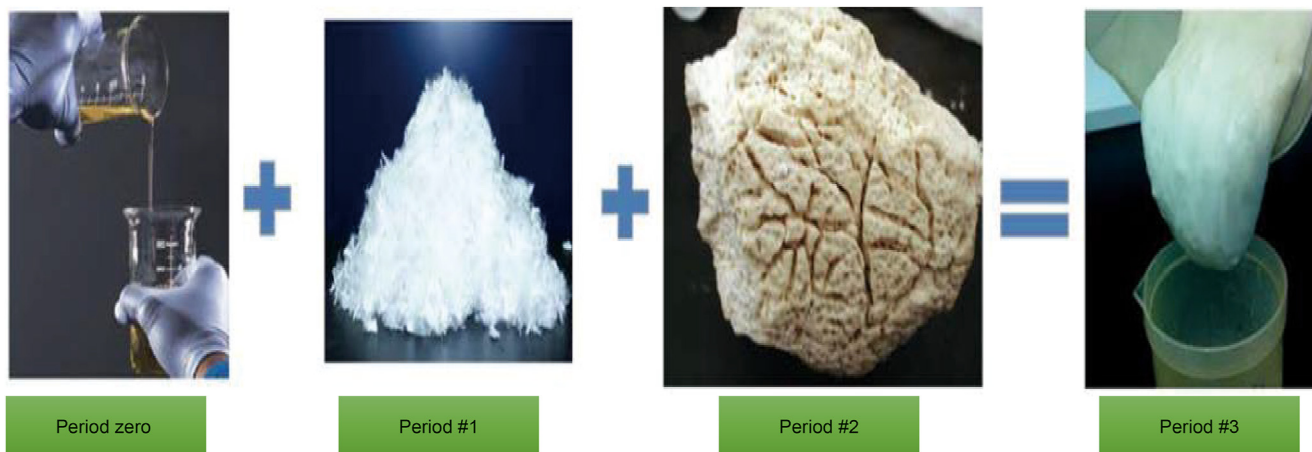

Fig. 22. Sequence associated with the deployment of a fiber viscoelastic self-bleeding acid (FLVSDA) in a fractured carbonate reservoir. The FLVSDA diverts the acid treatment due to its high viscosity and high fiber concentration (modified after Pooniwala et al., 2022).

Al$_2$O$_3$-acid, and ZnO-acid were used. Their first hybrid, nano-acid fluid (HNAF-1) with SiO$_2$ (NP) and HCl at concentrations of 0.4% and 6%, respectively, provided the greatest amount of oil displacement efficiency (DE), and about 16 % incremental oil recovery. For HNAF-2, Al$_2$O$_3$ (NP), and HCl at concentrations of 0.1% and 6%, respectively, DE was comparable using sequential and simultaneous techniques. For their HNAF-3 nano-fluid, ZnO (NP) and HCl at concentrations of 0.4% and 3%, respectively, with electrokinetics (EK) applied simultaneously improved oil recovery by 10% (Kim et al., 2020).

The interaction of engineered water with an enhanced sulfate ion content, together with a silica nanofluid, successfully altered the wettability of dolomite rock samples (with a diameter of 1.5 inches and a length of 55−70 mm) (Mahmoudpour and Pourafshary, 2021). An optimal engineered water and silica nanofluid concentration of 0.1 wt % was tested for use in the hybrid approach. The hybrid engineered water/nanofluid technology recovered 4.5% more oil than the optimal engineered water injection method used alone in core-flood testing. The NP in the injection fluid improved the IFT reduction and the production of microemulsions by reducing residual oil deposition on the rock surface (Mahmoudpour and Pourafshary, 2021).

A nano-encapsulated acid, ethylene diamine tetra-acetic acid (EDTA) has been utilized as a stimulating fluid, with 3-glycidoxypropyl-triethoxy silane SiO$_2$ (GPTS-SiO$_2$), and the results indicated a considerable rise in permeability associated with a minor increase in porosity (Petrakov et al., 2021).

NP tests with a gelled acid system showed that, following the acid breakout from the high permeability core, a single-stage nanofluid (1.25 vol% Si NP (7 nm in diameter) with a 9% cross-linker B and 15% HCl) could successfully redirect the acid into the low-permeability portion of a core (Kalgaonkar and Baqader, 2022). The tests and model demonstrated that when NP dispersions are injected into a preexisting wormhole, gelation can occur, although it took longer than in batch studies. NP-based gelled-acid systems offer the potential to replace polymer-based gelled acid systems. Such systems have been tested to function with up to 28 % HCl at temperatures up to about 150 °C with minimal viscosity at the surface, making them easy to pump. The pH of the fluid changes from acidic to basic as the acid is spent during reactions with the carbonate formation (Kalgaonkar and Baqader, 2022). The novel system's gelation phenomena was regulated by raising pH. As the pH increased above a value of 1, the NP gel caused the acidic fluid to congeal. As the pH increased above a value of 4, the NPs lose their capacity to gel and the fluid viscosity reduces to pre-gelation levels, allowing for smooth post-treatment flow return (Kalgaonkar and Baqader, 2022). Fibers offer an excellent temporary plugging mechanism for small fracture widths. On the other hand, fibers are less effective at plugging larger fracture widths. As a result, fibers are frequently employed in conjunction with other diverting agents to increase plugging strength. Furthermore, when deploying fibers, displacement needs to be sufficiently decreased to allow the fibers to easily accumulate and snag in the fractures.

5. Recommendations for future research

A wide range of possible technologies and methods of directional acid treatments are now available. These effectively solve a range of reservoir problems, particularly by increasing the hydraulic conductivity of low-permeability reservoir sections and enhancing production/injection rate profiles in specific wells. Although hydrochloric acid treatments are relatively cheap to implement their effectiveness can be limited. Both economic and technological factors need to be taken into account when customizing an appropriate acidization treatment to suit a specific reservoir target. Further research is required to improve the performance and reliability of acid treatments, including:

(1) The main purpose of acid treatment is to create a network of highly permeable channels in the target formation zone(s) by rock dissolution. To achieve the most efficient and optimal shape and size of wormholes, many factors must be considered, particularly the direction of acid flow into the target formation zones. This is especially important for wells penetrating several productive horizons with very different porosity/permeability properties. There is a risk that the acid treatment injected will escape into the more absorptive (high permeability) zones, leaving the low-permeability zones, most in need of stimulation, untreated. Another important control factor is the regulation of the reaction rate of the acid with the rock. More field-scale research is required to improve understanding of how best to control these key factors.

(2) To increase the depth of formed channels after acid treatment, it is necessary to slow down the reaction rate of the injected acid treatment with the carbonate formation. This requires modifying the acid composition so that it retains acidity for as long as possible. Hydrochloric acid has a high carbonate dissolution, so it is the most common reagent for bottomhole zone treatment in carbonate reservoirs. More research is required to investigate the benefits of alternative acid compositions.

(3) There are two characteristics that limit the use of hydrochloric acid: 1) reaction rate, and 2) corrosivity of tubulars and formation fluids. These problems can be at least partially mitigated by using emulsified acid compositions, such as

(a) Multi-stage stimulation
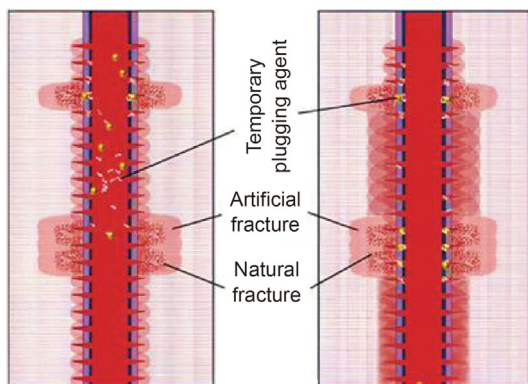
(b) Fracture network in the lateral direction
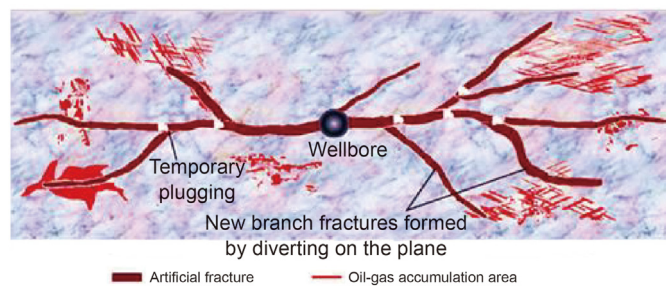
Fig. 24. Diverting mechanism of fibers deployed in combination with acid fracturing: (**a**) stimulation in many stages with vertical temporary plugging, (**b**) a lateral fracture network is formed by temporarily blocking and redirecting stimulation fluid flow (modified after Xiong et al., 2018).

Table 8
Laboratory tests of acid stimulation treatments involving fibers as a temporary blocking agent.

Fiber type	Chemical composition	Properties of the core	Outcomes	Source
Degradable gelled-acid system containing fibers	20 wt% HCl, acid-gelled fiber with different concentration (0.1, 0.2, 0.3, 0.4, 0.5 wt%); 0.8 wt% polyacrylamide; 0.3 wt% corrosion inhibitor	Sichuan and Tarim Basin of China (marine carbonate hydrocarbon reservoir). Depth: 7500—8800 m; reservoir temperature: 155—185 °C; reservoir pressure: 86—101 MPa; permeability of the upper and lower intervals: 1000 and 3 mD; porosity: 2 %.	At temperatures of 60, 80, and 100 °C, the increase in fiber concentration gradually increased the apparent viscosity of the acid system, while the increase in apparent viscosity decreased with increasing temperature. At the fiber degradation temperature (120 °C), the fibers could not significantly affect the apparent viscosity. However, when the formation temperature is below the fiber degradation temperature, increasing the fiber concentration could increase the viscosity of the acid. When the formation temperature was higher than the fiber degradation temperature, it was not appropriate to increase the concentration of fibers to increase the viscosity of the acid. The process of dissolving the fiber in gelated acid went through the stages of non-dissolution, surface dissolution, dissolution and grinding, and complete dissolution at an increase in temperature, which in different ways affected the viscosity of the gelatinized acid.	Gou et al. (2021)
Viscoelastic self-dissolving acid with fibers (FLVSDA)	175 lb per 1000 US gallons of FLVSDA; 15 wt% HCl	Carbonate reservoir stimulated by acidic hydraulic fracturing. A new high-temperature decomposable fiber with two distinctive features was developed. First, a modified polymer with a highly ordered structure is used, resulting in a higher melting point and increased thermal and hydrolytic stability compared to modern medium-temperature fibers. Second, its morphology is corrugated, which provides better dispersion of the material and clogging efficiency at higher concentrations. Laboratory tests were conducted to compare degradation and stability in neutral and acidic environments to reproduce real acid treatment conditions. Jumper tests were also conducted in the slit geometry to characterize the effectiveness of the removal of suspensions containing fibers. Finally, the material was tested in fields with temperatures ranging from 43 to 165 °C.	Fiber integrity and stability were distinguished by the characteristics of the high-temperature degradable fiber and medium-temperature fiber at temperatures above 135 °C. The critical efficacy point of high-temperature degradable fiber was achieved after 6 h of exposure at 143 °C at 100% 15% HCl with a concentration of 175 pounds per 1000 US gallons. On the other hand, medium-temperature fiber remained stable for less than 2 h under the same test conditions. The high-temperature degradable fiber demonstrated similarly improved outlet efficiency when tested in more antagonistic environments such as 50% waste acid.	Leal Jauregui Jairo et al. (2010)

those based on acid/oil emulsions in carbonate reservoirs (Chacon and Pournik, 2022; Gozel et al., 2021; Hao et al., 2019; Jafarpour et al., 2017; Rabie and Nasr-El-Din, 2015; Xu et al., 2020). Emulsified acids expand their depth of penetration into the target formation and reduce the corrosivity of the injected fluid. More research is required to optimize the formulation of emulsified acids.

(4) Injecting acids into target zones does not guarantee their efficient consumption and influence throughout the formation. In many formations, a network of preferential wormhole channels is created rather than consistent cavernous porosity. Stimulation of water-saturated intervals tends to be ineffective. Consequently, there is a need to develop selective agents capable of blocking water-saturated zones and some wormholes in order to focus the stimulation on low permeability, oil-saturated zones. More research is required to develop cheap and effective temporary blocking agents.

(5) Foam-forming agents must rapidly generate stable foam in the formation during gas injection. They also need to be chemically compatible with formation fluid and rock compositions, not degrade reservoir permeability, have anti-emulsion properties, and be stable at formation temperatures. There is scope to improve existing foaming agents with respect to these properties.

(6) The historical performance of acid treatments has been mixed, with many not delivering positive production benefits, and the successful treatments rarely improving production by more than 30%. The low cost of implementing basic acid treatments has inhibited research into finding more costly improvements. If one acid treatment delivers poor results it is often easier and cheaper to conduct another similar treatment rather than spend time and cost to improve the acid treatment design. More field testing of different acid treatment designs are required to explain the poor performance of basic treatments in some reservoirs.

(7) *In-situ* acid gelled systems contain a polymeric gelling agent, an iron-based crosslinking agent, a degradation chemical, and additional additives, with acid serving as the base fluid. Treatments involving polymer additives, can cause formation damage for a variety of reasons, including undissolved polymer residues. Additionally, when gelled-acid reacts with the formation, the iron-based crosslinker components can cause scale and/or sludge formation, resulting in formation damage and reduced hydrocarbon productivity. Further research is required to avoid these problems, such as using NP or JNP additives.

(8) Acid-treatment formulations are typically only suitable for temperatures up to 160 °C. At higher temperatures experienced in wells drilled into deep carbonate reservoirs, the acid-rock reactions tend to occur too rapidly, are only effective over short distances into formation, and are associated with high corrosion rates. To address these problems, further research is required to develop acid formulations tailored for high-temperature, deep carbonate reservoirs. Such

Table 9
Field trials of acid stimulation treatments involving fibers as a temporary blocking agent.

Fiber type	Chemical compositions	Reservoir properties	Outcomes	Source
Self-disintegrating synthetic fiber J595 (degradable fiber)	VES in a mixture of HCl (15%) with degradable fibers (combination of self-decaying synthetic fibers J595 and viscoelastic polymer-free acid VDA)	South Gavar field (Saudi Arabia), Huff heterogeneous carbonate reservoir. Well A: Depth 4109.3 m, permeability 1 mD, porosity 11%, reservoir thickness 30.48 −60.96 m, reservoir temperature 148.9 °C, reservoir pressure 57.84 MPa. The produced gas contains less than 1% H_2S and CO_2. Well B: Depth 3725.6 m, permeability 13 mD, porosity 10%, reservoir thickness 30.48 −60.96 m, reservoir temperature 148.9 °C, reservoir pressure 33.78 MPa. The produced gas contains less than 4% H_2S and CO_2.	During the field trial, two liquids were squeezed through a 2-mm hole, imitating a natural fracture channel. After a short time, the composition system (VES in a mixture of HCl (15%) with degradable fibers) was able to form a stable crust on the walls of the channel. When testing the VDA system without fiber, a similar effect was not obtained, and the liquid was absorbed by the reservoir at a constant rate.	Leal Jauregui Jairo et al. (2010)
Self-decaying synthetic fiber	VES in a mixture of HCl (15%) with degradable fibers (90 lb/ 1000 gallon), ammonium chloride, and nitrogen to accelerate the purification of squat-gin.	Huho-Tecominoacan (Mexico) field. Depth: 5000 m, reservoir temperature:120−160 °C, reservoir pressure: 22.8 MPa, the permeability of the upper and lower intervals: 1000 and 3 mD, respectively, porosity: 5% −8%.	The acid treatment was pumped into a well without direct control of the flow of liquid into a particular interval ("head-on" method). Sequential injections were made of 30 m^3 (7800 US gallons) of aromatic solvent to clean the perforation channels, 60 m^3 (15,600 US gallons) mixture of HCl and formic acid, 10 m^3 (2600 US gallons) MaxCO$_3$ acid with a fiber concentration of 90 lb/1000 US gallons (11 kg/m^3) and 2 m^3 (520 US gallons) of ammonium chloride buffer solution. The pumping speed ranged from 8.2 to 15 barrels per minute (from 1.3 to 2.4 m^3/min). The last injection phase contained nitrogen to speed up the clean-up of the well. Oil production began within 3 days of the treatment. The initial oil flow rate was 3000 barrels per day (480 m^3/day). Three months later, the average oil flow rate stabilized at 1600 barrels per day (250 m^3/day (flow rate was 203 m^3/day)).	Asiri et al. (2013)
Self-decaying synthetic fiber	VES in a mixture of HCl (15%) with degradable fibers (90 lb/ 1000 gallons). Mutual solvents − these are chemicals containing both water-soluble and water-insoluble compounds, diesel fuel for washing can dissolve.	Pre-salt deposits in Brazil (phenolic carbonate layers). Depth: 4500−6500 m, reservoir temperature: 60−133 °C, permeability: from less than 0.001 mD to several tens of mD, porosity: 5%−18%, layer thickness: 300−400 m.	The treatment involved the injection and deviation of acid into the matrix at depths of 4500−4570 m. Determined by simulation, the treatment required a "head-on" injection of 13 packets of solution each in volumes of 12.6 m^3. These included a 15% solution of HCl, a liquid VDA, and a liquid MaxCO$_3$ acid with a fiber concentration of 12−14 kg/ m^3. Before injecting acid, a mixture of saline solution and HCl with a mutual solvent - monobutyl ether was pumped into the well. Following the treatment, another packet of HCl with a mutual solvent was pumped into the well, after which the well was washed with diesel fuel. The pump speed varied from 0.8 m^3/min during injection of MaxCO$_3$ acid packets to 1.6 m^3/min during HCl injection and up to 3.2 m^3/min during injection of VDA deflective solution.	Asiri et al. (2013)
Self-decaying synthetic fiber	VES in a mixture of HCl (15%) with degradable fibers.	Tengiz field (Caspian Sea, Kazakhstan). The reservoir is characterized by a fractured carbonate with porosity and permeability ranging from 3% to 20% and 1 to 10 mD, respectively. The reservoir pressure is 51.7 MPa, the reservoir temperature is 104−116 °C. The productive part of the reservoir is about	The treatment used the MaxCO$_3$ acid system injected in 2−5 stages. Initially, a solution of 15% HCl was injected to contact the most permeable zones. The VDA system was pumped into the well, acting as a buffer to prevent acid contact with fibers. A diversion system was then established by injecting fibers and a VDA system, pumped in one after the other. Intertwined fibers blocked the flow of the injected acid into the	Manakhayev et al. (2018)

Table 9 (*continued*)

Fiber type	Chemical compositions	Reservoir properties	Outcomes	Source
		800 m thick, the depths to the top of the productive layer are in the range from 3800 m to 5500 m. Reservoir gas is characterized by very high H_2S of about 14%.	most permeable zones. The presence of the VDA system enhanced acid deflection. In the final step, additional HCl is pumped and deflected by the fibers and the VDA system to contact the less permeable carbonate zones. After processing, the flow rate was 1122.45 m^3/day, i.e., 2.5 times higher than before the treatment.	
Self-decaying synthetic fiber	6% ClearFRAC XT (polymer-free fracturing fluids), VDA, DGA420 (acid) MaxCO$_3$-20 (VES in a mixture of HCl (15%) with degradable fibers.)	Jurassic reservoirs (Kuwait). The Marratus Formation (incorporating the Hittang to Toark intervals) is formed of limestones and dolomites displaying a combination of matrix porosity as well as low-porous fractures. The dolomite layers contain the best porosity (up to 12%).Reservoir depth is 5117.6 m, average reservoir temperature is 132.2 °C, and reservoir pressure is in the range of 68.95—82.74 MPa. The content of H_2S in the gas in these formations varies between 0.5% and 10%, averaging 800 ppm.	During the main acid treatment, high pressure of 95.15 MPa was maintained at the injection surface, accompanied by a large number of fractures. The results after acid fracturing showed a significant increase in productivity index (PI) and the production rate improved three to four-fold.	Al-Otaibi et al. (2020)

formulations need to achieve friction reduction, high-temperature corrosion inhibition, temperature and shear resistance, and acid-rock reaction rate reduction.

(9) The most likely causes of low efficiency of acid treatments in target formation zones in carbonate reservoirs include: acid penetration into previously depleted intervals with higher water saturation; quick exhaustion of the conventional acid composition in the formation; intensive dissipation of the acid composition through induced fractures formed in the matrix during the acid fracture stimulation; incorrect selection of the acid composition injection, which is unable to create extensive dissolution channels in the formation. To avoid such outcomes, it is important to conduct rigorous laboratory tests with rock samples from the target formation to optimize the acid-rock reaction rate.

(10) The success of deep acid penetration is ensured by reducing the activity of the acid formulation. A number of reagents are available that reduce the reaction rate and allow the acid to penetrate further into the formation. Acid-rock reaction rates can be slowed by thickening the acid mixture with additives, emulsifying it, gelling it or generating acid in the formation. More research is required to tailor these methods, and identify new acid retardants, to suit specific sub-surface conditions, particularly deep high-temperature formations. More work on organic acids and chelate compounds is required.

(11) Further research is required into how best to prevent colmatation (clogging) of target formations during acid treatments. Currently, there are no methods for calculating the risks of deposition of heavy components from formation oil (e.g. asphaltenes, heavy metals) during acid treatments based on oil compositions, formation geology, and/or physical subsurface conditions.

(12) A key disadvantage of hydrochloric acid use for sub-surface acid treatments is its corrosiveness causing precipitations when coming into contact with formation fluids. Certain additives can reduce the corrosivity of an acid solution.

Viscoelastic surfactants (VES) can be effective in this regard. The beneficial properties they exhibit are due to the aggregation of surfactants with the formation of micellar structures with viscoelastic characteristics at low concentrations of surfactants. More research is required to better understand the processes involved and extend VES applications.

(13) In polymer-based *in-situ* gelled acid formulations composed of HCl, a polymer (typically, silicate or polyacrylamide), crosslinker (typically, Fe^{3+}, Al^{3+}, or Zr^{4+}) and breaker (sodium erythorbate, to oxidize ferric to ferrous ions and thereby reduce viscosity) are usually involved. The breakers are pH sensitive and specifically control the crosslinking of polymer chains in the solution. As with VES, the interaction of acidic solutions with carbonate rocks increases pH. Raising the pH to a certain level activates the crosslinking of polymer chains in the injected solution and increases its viscosity. As a result, wormhole growth in high-permeability zones can be inhibited, and the injected treatment is redirected from the high-permeability zones to the low-permeability zones. Despite these benefits, recent laboratory studies have shown that the use of polymers and crosslinkers (usually consisting of Fe^{3+} and Zr^{4+}) can cause damage during the reflux step of the acid treatment process. Further research is required to develop more reliable polymer-crosslinker systems.

(14) Further investigations focused on the use of various types of NP with acids at different concentrations are required, especially to better understand their influences on acid treatments at high temperatures and high pressures.

6. Summary and conclusions

The findings and conclusions of this review can be summarized as:

- Self-diverting acid (SDA) systems can be based on foam, emulsion acid, gelated acid, visco-elastic surfactants (VES), cross-

linked polymers, and fibers. The main advantage of an SDA is that, it does not colmatize the pore space of the reservoir.

- Key factors influencing thermal-foam-acid treatment are: the interaction of magnesium and hydrochloric acid; the interaction of solutions of heat- and gas-forming agents; and, the injection of hydrochloric acid from the wellhead. Thermal-foam-acid treatment systems tend to involve the sequential injection of aqueous solutions containing heat- and gas-forming agents with an initiator.

- Cationic surfactants are favored to stabilize emulsified acids. They typically involve corrosion inhibitors to mitigate tubular corrosion. Particulate agents are sometimes used in acid emulsions to achieve robust thermal stability and reduced resistance to injection. Ensuring all the additives interact effectively remains a key challenge for successful field-scale deployments of emulsified acid systems.

- Pumping a gel solution instead of a conventional acid solution typically generates a deeper and longer-lasting reservoir treatment. It does so by reducing the rate of reaction with the carbonate formation. The gel penetrates predominantly into the high permeability zones of the formation. In high-temperature formations, where conventional acid solutions quickly lose their activity, gels are most effective. The retention of high gel viscosity makes them particularly useful for acid fracturing in high-temperature reservoirs. Over time, the gel composition decomposes, which solves the problem of removing spent acid from pores and fractures in the formation. The application of gels is currently limited to reservoir temperatures up to 135 °C. Moreover, by using a combination of zwitterionic and anionic surfactants a minimum viscosity of the initial acid solution can be guaranteed. This is a consequence of the protonation of the zwitterionic surfactant in the acid medium, causing it to act as a cationic surfactant. It is beneficial as it prevents the interaction of the components with each other before they react with the carbonate formation. This behavior also simplifies the injection process.

- The inclusion of tiny fibers as an additive to acid treatments is well established. Originally fibers were used as a flow-back-prevention additive, but they also act as effective matrix diverting agents and fracture acidizing agents. Employing fibers as temporary plugging agents has the following limitations: (1) most temporary-plugging equipment used has a low pressure-bearing capability, with a maximum plugging pressure of less than 6.9 MPa (1000 psi); (2) the influence of fracture morphology on temporary plugging is rarely taken into consideration; and (3) the majority of individual fractures are small with limited accessible lengths, with a length averaging about 6 cm.

- Nanoparticles (NPs) combined with acid to treat carbonate reservoirs substantially change wettability resulting in a positive impact on resource displacement efficiency. Silica NPs have so far achieved the highest oil displacement efficiency associated with such treatments.

Declarations of competing and/or financial interests

The authors confirm that they have no known conflicts of interest regarding the information provided in this study, neither have they received financial support for this work, which has influenced its content in any way.

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Nomenclature

Acronyms

AC	Acid compositions
AHF	Acid hydraulic fracturing
Al_2O_3	Aluminum oxide
HNH_2SO_3	Aminosulfonic acid
PASD	Amphoteric polyacrylamide
NaOA	Anionic surfactant
$CaFe(CO_3)_2$	Ankerite
$CaCO_3$	Calcite
Ca^{2+}	Calcium
$CaCl_2$	Calcium chloride
CO_2	Carbon dioxide
CMC	Carboxymethyl cellulose sodium
CMHPG	Carboxymethyl hydroxypropyl guar
CNF	Cellulose nanofiber
CNEVFF	Cellulose nanofiber enhanced viscoelastic fracturing
$PbCO_3$	Cerussite
CTAB	Cetyltrimethylammonium bromide
CA	Citric acid
COP-2	Clay stabilizer
HiWAY	Cyclic injection of proppant with fibers
DE	Displacement efficiency
$CaMg(CO_3)_2$	Dolomite
EK	Electrokinetics
EOR	Enhanced oil recovery
EDTA	Ethylene diamine tetra-acetic acid
ERD	Extended reach drilling
FiberFRAC	Fiber reinforcement technology for hydraulic fracturing fluid
FOCT	Fiber-optic-enabled coiled tubing
DR800	Friction reducer (anionic polyacrylamide)
GQA	Gemini quaternary ammonium salt cationic surfactant
GPTS	3-Glycidoxypropyl-triethoxy silane
HMB	High molecular weight compounds
HT	High-temperature
HCl	Hydrochloric acid
HPAM	Hydrolyzed polyacrylamide
HLB*	Hydrophile-lipophile balance
HLB	Hydrophilic surfactant
HEC	Hydroxyethylcellulos
HPG	Hydroxypropyl guar gum
EIOR	Improved oil recovery
IFT	Interfacial tension
Fe^{2+}	Iron
$CaMn(CO_3)_2$	Kutnagorite
LB	Lipophilic surfactant
LT	Low-temperature
$MgCO_3$	Magnesite
Mg^{2+}	Magnesium
GF−15 MPS	Mixture of alkyldimethylbenzonium chloride, tertiary amine, stabilizer and glycolylic solvent
HNAF	Nano-acid fluid
NP	Nanoparticles
N_2	Nitrogen
Alkonat L90	Nonionic surfactant
O/W	Oil-in-water
PLA	Polylactic acid fiber
PVBT	Pore volume of breakthrough
$MnCO_3$	Rhodochrosite
J595	Self-disintegrating synthetic fiber
SDAS	Self-diverting acid system
SFDD	Sequenced fracture degradable diverter system

SiO$_2$	Silicon dioxide
ZnCO$_3$	Smithsonite
NaCl	Sodium chloride
SDBS	Sodium dodecyl benzene sulfonate
NaOH	Sodium hydroxide
SrCO$_3$	Strontianite
TGF-1	Surfactant transforming growth factor
IF9-12	Surfactant-neonol
EDTA-Na4	Tetrasodium salt of ethylenediaminetetraacetic acid
MaxCO$_3$	The self-diverting acid degradable fiber diversion acid system
TFAT	Thermo-foam acid treatment
VDA	Visco-elastic polymer-free acid
FLVSDA	Viscoelastic self-dissolving acid with fibers
VES	Viscoelastic surfactant
W/O	Water-in-oil-type
EXTELINT®	White fibers
ZnO	Zinc oxide

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