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Research Article

Application status and research progress of shale reservoirs acid treatment technology

Li Nianyin [a], Dai Jinxin [a,*], Li Jianhui [b], Bai Fengjun [c], Liu Pingli [a], Luo Zhifeng [a]

[a] *State Key Laboratory of Oil and Gas Reservoir Geology and Exploitation, Chengdu 610500, China*
[b] *Oil & Gas Technology Research Institute of PetroChina Changqing Oilfield Company, Xi'an 710018, China*
[c] *Changqing Downhole Technical Operation Company, CNPC Chuanqing Drilling Engineering Co. Ltd., Xi'an 710018, China*

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Abstract

In recent years, shale oil and gas development has been thriving in China. However, the shale oil and gas production always suffers a rapid decline. Based on the analysis of a large amount of former theories and experiences, a summary of acid treatment stimulation methods in shale oil and gas is presented, and the acid stimulation mechanism is analyzed. The mainstream technique in acid treatments includes: acid wash, matrix acidizing, prop fracturing with acid preflush, and multi-stage alternate-inject acid fracturing. The main stimulation mechanism of acid treatment can be summarized into 3 categories: a) the influence on shale matrix, namely the acid-induced increase of porosity and permeability, and reduce of wetting property of shale; b) the influence on rock mechanical properties, namely shale brittleness and toughness, and even Young Modulus to some degree; c) the influence on fractures' conductivity, caused by the fact that acid dissolves calcite-enrichment area in priority, and then increases roughness on fracture surface. In room temperature and atmospheric pressure, acid reduces fractures' conductivity, while in pressurized condition, the acid-soaked fractures' conductivity is higher than the conductivity of non-acid-soaked fractures. These knowledges would provide useful reference for furthering stimulation techniques and processes in shale oil and gas development.

Keywords: Shale reservoirs; Acid treatment technology; Stimulation; Stimulation mechanism; Research status

With the ever increasing demand for energy and depleting conventional reservoirs, the oil and gas industry is more and more dependent on the commercial development, and prolonging production from unconventional shale counts. The exploitation of shale oil and gas resources has great benefits from the progress of horizontal drilling techniques combining with hydraulic fracturing. Slick water is the most commonly used fracturing fluid, which may be adopted to achieve maximum contacted reservoir surface area, known as stimulated reservoir volume (SRV) [1].

However, there are still many challenges in the process of production, the most important challenge is that many of the wells suffer drastic decline in production over the initial one year period [2]. The reason is due to a) the proppants are in uneven distribution. The complex fracture network is created by slick water. In medium, there are many microfractures whose width is mainly between 10^{-6} cm and 10^{-1} cm. Because they are too small to be accessed by most common proppants, such as 40–70 or 100 mesh sand, and the proppant carrying ability of slick water is low (maximum is 2 lbs/bbls), therefore, when reservoir pressure declines as oil and gas produced from the formation, the declining pore pressure leads to increase of effective closure stress. As a result, microfractures are not able to support the increasing effective closure stress mounts. This condition limits the producible reservoir volume to the primary fracture, leading to a steep decline in production [3–10]; b) proppant fatigue due to stress cycling and digenesis [11]. Therefore, we need find a way to keep these unpropped fractures and microfractures open. Acid fracturing, as an alternative technology for hydraulic propped

* Corresponding author.
E-mail address: daijxin1987@126.com (Dai JX.).
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fracturing, can be a potential solution to improve the productivity of microfractures in carbonate-rich shale.

It is widely believed that acid treatment technology is basically invalid in shale oil and gas stimulation, because: a) although shale contains some acid soluble minerals, such as calcite and dolomite, their distribution is very uneven in shale, which induces difficulties in creating successive channels with conductivity after acid dissolve these minerals; b) the mineral composition of shale is of great variety, thus the effect of acid stimulation becomes difficult to estimate. Therefore, to alter stereotypes for only use of hydraulic fracturing in shale oil and gas stimulation, this paper introduces the application of acid treatment technology in shale oil and gas stimulation and discusses its stimulation mechanism in detail. It can be concluded that, acid treatment technology has a great application prospect and provides a new stimulation idea for shale oil and gas reservoir stimulation.

1. Application status of acid treatment technology in shale oil and gas reservoir

1.1. Downhole acidic scale removal and matrix acidizing

Acid was initially used to stimulate shale oil and gas well for the purpose of removing precipitated carbonate and of well stimulation. Honolulu Oil Corp., operator for the Antelope Shale Zone Unit, Buena Vista Hills, initiated an acid treatment program, Oct. 23, 1956. 35 wells were treated with acid, and yielded a net increase oil of 1390B/D and gas increase of 3265 Mcf/D [12].

Except for downhole acidic scale removal, matrix acidizing is also an effective stimulation method. In matrix acidizing, acid is injected into the near-wellbore reservoir, and then it removes the blockage in the near-wellbore reservoir, such as precipitated carbonate and mud pollution, and dissolves some carbonate rock which is packed in natural fracture, and etches the fracture face. Correspondingly, the near-wellbore reservoir conductivity is recovered or even increased, and oil and gas production increase. There are many successful cases of matrix acidizing in shale oil and gas reservoir [13—16]. One area of successful matrix acidizing was in the Monterey N/NA shale at Elk Hills, Nabil EL Shaari [13] believed that the success is due to a) improving the wellbore connection into naturally-fractured calcareous intervals, and b) the impact of reducing skin across a thick production interval.

1.2. Pad-acid-hydraulic fracturing technology

In S.E. Oklahoma Woodford shale frac job, injection of thin frac treating fluid into shale results in high pumping pressure and low injection rate, so volumes of various types of acid systems are pumped with surprising results [17]. The job plot is shown in Fig. 1. Likewise, J. Fontaine [18] introduced that Marcellus shale gas frac job have similar procedure. Pad acid not only can obviously reduce initiation fracture pressure, but also can remove the pollution in near wellbore. Therefore, this hydraulic fracturing method with pad acid is widely applied in shale oil and gas stimulation [19—23].

1.3. Multistage alternating injection proppant-carrying acid fracturing

Acid treatment technology is not just limited to acidic scale removal, matrix acidizing and pad-acid-hydraulic fracturing. Currently, multistage alternating injection proppant-carrying acid fracturing technology has obtained successful application, which combines the advantages of acid fracturing with hydraulic fracturing, not only can remove the near wellbore pollution, improve the microfracture conductivity, but also can release more adsorbed gas.

In one reported case, two of six Caney shale wells were treated in McIntosh County, Oklahoma using acid. Verbal reports indicated a two-fold higher initial production (IP) in the treated vs. untreated wells. In another case in Coal County, Oklahoma, two Woodford shale wells treated with acid were reported to flow up casing for a two-week period in an area not normally capable of flowing without assist. Table 1 illustrates a Woodford shale pump schedule incorporating the use of 3% HCl. Up to 280,000 gallons of 3% acid, or 30% by volume of total fluid pumped, have been placed in a Caney shale well [17].

Williams Production Co. experimented with acid in the Caney shale [8]. Initial production from their first attempt was exceptional compared to previously completed wells. The stimulation effect in the well lasted past 200 days.

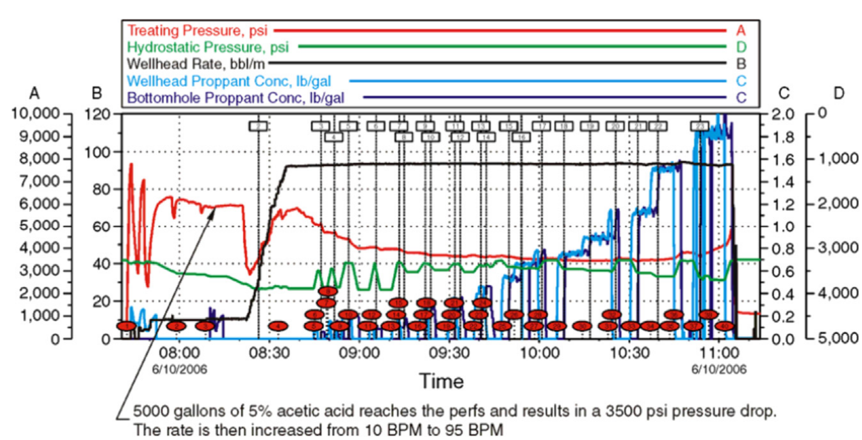


Fig. 1. Job plot from S.E. Oklahoma Woodford shale frac job showing dramatic/unexpected pressure drop as reactive fluid hits the perfs.

Table 1
Pump schedule: Woodford Shale frac treatment.

No.	Stage	Vol., gal	Fluid	Conc., lbm/gal	Proppant
1	Acid	4000	15% HCl acid		
2	Pad	26400	Pad and flush		
3	Sand slug	5000	Treated water	0.1	Premium Brown-30/70
4	Pad	26400	Pad and flush		
5	Sand slug	5000	Treated water	0.15	Premium Brown-30/70
6	Pad	26400	Pad and flush		
7	Sand slug	5000	Treated water	0.2	Premium Brown-30/70
8	Pad	26400	Pad and flush		
9	Sand slug	5000	Treated water	0.25	Premium Brown-30/70
10	Pad	26400	Pad and flush		
11	Sand slug	14240	Treated water	0.1	Premium Brown-30/70
12	Acid	7120	28% HCl acid cut on the fly to 3%		
13	Sand slug	14240	Treated water	0.19	Premium Brown-30/70
14	Acid	7120	28% HCl acid cut on the fly to 3%		
15	Sand slug	14240	Treated water	0.28	Premium Brown-30/70
16	Acid	7120	28% HCl acid cut on the fly to 3%		
17	Sand slug	14240	Treated water	0.37	Premium Brown-30/70
18	Acid	7120	28% HCl acid cut on the fly to 3%		
19	Sand slug	14240	Treated water	0.45	Premium Brown-30/70
20	Acid	7120	28% HCl acid cut on the fly to 3%		
21	Sand slug	14240	Treated water	0.55	Premium Brown-30/70
22	Sand slug	14240	Treated water	0.64	Premium Brown-30/70
23	Sand slug	14240	Treated water	0.73	Premium Brown-30/70
24	Sand slug	14240	Treated water	0.82	Premium Brown-30/70
25	Sand slug	14240	Treated water	0.9	Premium Brown-30/70
26	Flush	3656	Pad and flush		

2. Research progress of shale oil and gas reservoirs acid treatment technology

As acid treatment technology has achieved an initial success in shale oil and gas reservoirs, some researchers start studying stimulation mechanism of acid treatment. Table 2 illustrates a core composition incorporating the use of main three researchers. Based on these research results of generalizations, we discover that stimulation mechanism researches mainly concentrate on three aspects: the effect of acid on shale matrix, the effect of acid on shale mechanical properties, the effect of acid on fracture conductivity. These research achievements have great and profound meanings for shale oil and gas acid stimulation [17,24–35].

2.1. Effect of acid on shale matrix

(1) Acid increases porosity and permeability of shale matrix. The increase of matrix porosity depends on acid strength and acid rock contact time. Acid soluble minerals are dissolved by acid, and then many microfractures and holes are exposed from shale matrix and the size of original pores grows, therefore, matrix porosity increases. The increase of porosity may be not proportional to the content of acid soluble minerals. This may be due to the different distribution of acid soluble minerals in shale cores. After acid soluble minerals are dissolved, some acid insoluble minerals may move and block pores, and thus decrease the porosity. The improvement in sample permeability was well observed after exposure to low pH solutions, as the fluid in the samples were recognized as well connected in visual lines across the sample. Therefore, improper acid strength and acid rock contact time not only cannot increase matrix porosity, but also may decrease matrix porosity, so we should choose proper acid strength and acid rock contact time based on acid fracturing layer of interest.

(2) Acid reduces shale matrix contact angles and so increases oil recovery. The shale wettability was altered to strong water-wet by using low-concentration acid, and the values of the measured contact angles are well correlated with the achieved recovery factors as the rock wettability was altered. Oil recovery factors were enhanced by mineral dissolution and wettability alteration.

(3) Acid may enhance diffusivity of gas into the fracture network. Shale surfaces contacted with SRF (exposed micropores and increased surface area) show a significant increase in effective surface area and interconnection to adsorbed gas. The total amount of gas contained in the shale is believed to be composed of "free" and "adsorbed" gas [36]. The free gas is likely to be contained in: a) the micropores present in the bulk volume shale; b) the macropores contained in the thin laminations of chert, dolomite, calcite, and silt/sand present in most midcontinent shales; c) non-mineralized or non-healed natural fractures. The adsorbed gas is believed to be attached to the organic and clay material in the bulk shale.

While removal of an individual calcite or dolomite crystals does not always open a permeable path to micropores, it will

Table 2
A composition of core samples used by researchers.

Researchers	Core	Quartz %	Calcite %	Dolomite %	Pyrite %	Kaolinite %	Chlorite %	Mica %	Illite %	Etc. %
Samiha Morsy etc.	Eagle Ford	11.3	48		13	13	0	14		0.7
	Mancos	76	4.68		0	12	0	6.9		0.42
	Barnett	23	0		15	0	24	38		0
	Marcellus	71	15.7		3.4	0	0	9.6		0.3
Divyendu Tripathi etc.	Eagle Ford	3.9	48.8	11.9		2.1			6.4	26.9
Weiwei Wu etc.	Bakken	27.38	10.9	13.57		6.75			15.66	26.44

enhance the overall surface area of the treated shale. This surface area enhancement may be small in the micro scale, but may add up to significant area enhancement on the macro scale. This increase in surface area may increase the desorption and/or diffusivity rate of gas from the shale [37]. Scott Schad, in his work presented significant increases in calculated surface area due to micro-etching of the shale face in horizontal laterals and the resultant increase in estimated production efficiency due to increase in effective surface area of exposed shale [38]. The amount of gas produced by desorption should be directly related to the amount of surface area exposed, Fig. 2 illustrates a hypothetical response to gas diffusivity [17]. Acid may increase the surface area of a newly created hydraulic fracture. Conceptually, this is illustrated Fig. 3 and Fig. 4.

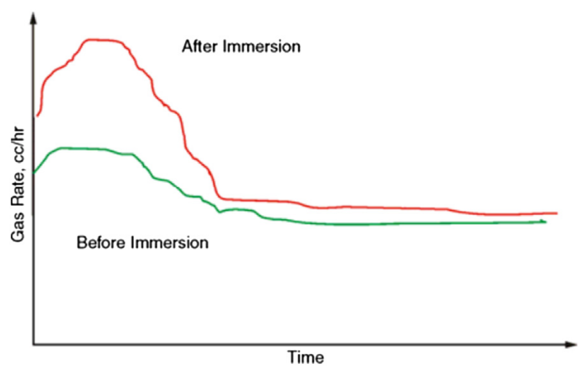


Fig. 2. Hypothetical conceptual plot of gas production from a shale sample before and after immersion of reactive fluid.

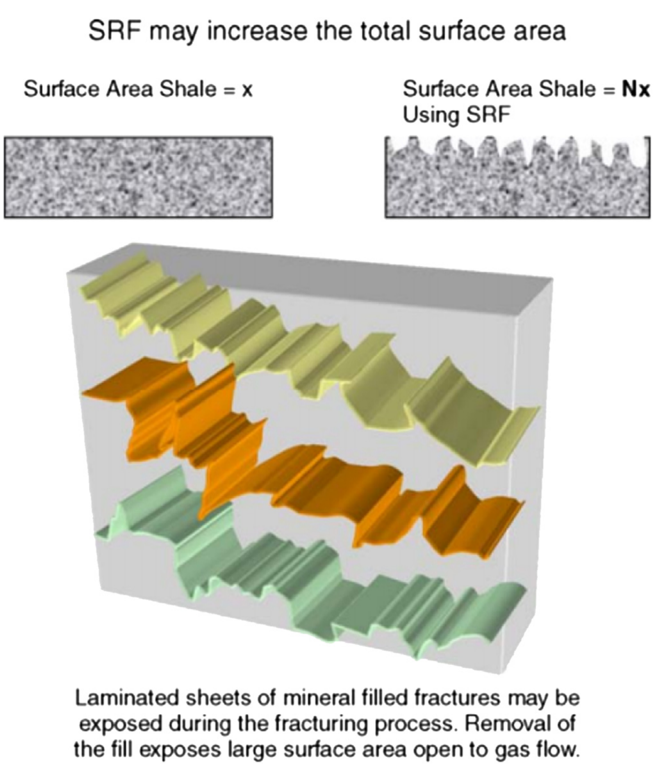


Fig. 3. Conceptual illustration of the effects of a SRF on shale fracture surface microstructure.

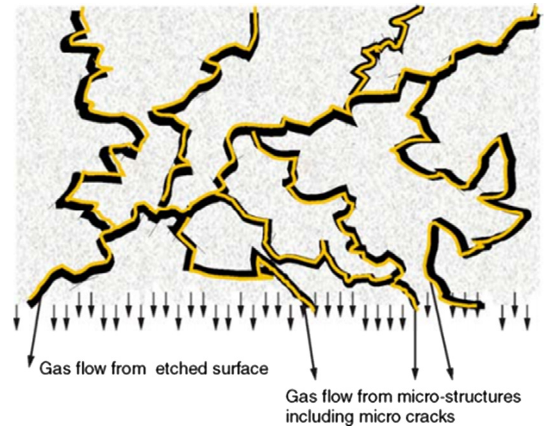


Fig. 4. Gas production from hydraulically fractured shale is believed to come from desorption and diffusivity from microporosity/fractures. SRFs may help remove acid-soluble minerals in the bulk shale as well as the mineral-filled fractures, thereby enhancing diffusivity of gas into the fracture network.

2.2. Effect of acid on shale mechanical properties

(1) Acid reduces shale hardness (Table 3). From Table 3, we can see that the rock hardness was significantly affected by low pH solutions, which resulted in 55–94% loss of its initial value using low pH solutions. While we also can see that the hardness did not continue lowering when pH 0.74 (3 wt% HCl) was used, instead, the measured hardness was about 30869 which was higher than those in lower acid concentrations. This change in hardness behavior when exposed to 3 wt% HCl solution might be due to iron precipitations that can add more hardness to the sample compared with lower HCl concentration solution. And the content and the distribution of acid soluble minerals also influence on the decreased degree of shale hardness.

(2) Acid reduces shale brittleness. Characteristics of the stress–strain curves for shale samples are presented in Fig. 5. The deformation of rock can characterize rock brittleness. From the change of characteristics of the stress–strain curves, we consider acid decreases shale brittleness.

(3) Acid may reduce shale Young's Modulus, but the loss degree in Young's Modulus is different for different shale samples. The research results of Morsy et al. show that low concentrations of HCl cause a huge loss in Young's Modulus ranging from 25 to 82%, while the research results of Weiwei Wu et al. show that no obvious changes were found in Young's Modulus by acid exposure (see Fig. 6). This happens because of different shale samples used by two researchers. Shale samples used by Morsy et al. are Eagle Ford shale, contain over 61% of acid soluble minerals, while shale samples used by Weiwei Wu et al. are Bakken shale, contain about 24% of acid soluble minerals. Therefore, we consider that the content of acid soluble minerals may cause the difference, because carbonate rock is of brittle rock [39], and the distribution of acid soluble minerals may also cause the difference.

Table 3
Measured hardness for Marcellus shale samples soaked in different solutions.

No.	Soaking fluid	pH of soaking solution	Conditions	Avg. BH (psi)
1	2 wt% KCl solution	7.4	represent initial conditions	68842.4
2	1 wt% HCl + 2 wt% KCl solution	1.21	After soaking	23583.6
3	2 wt% HCl + 2 wt% KCl solution	1.02	After soaking	4547.4
4	3 wt% HCl + 2 wt% KCl solution	0.74	After soaking	30869.6

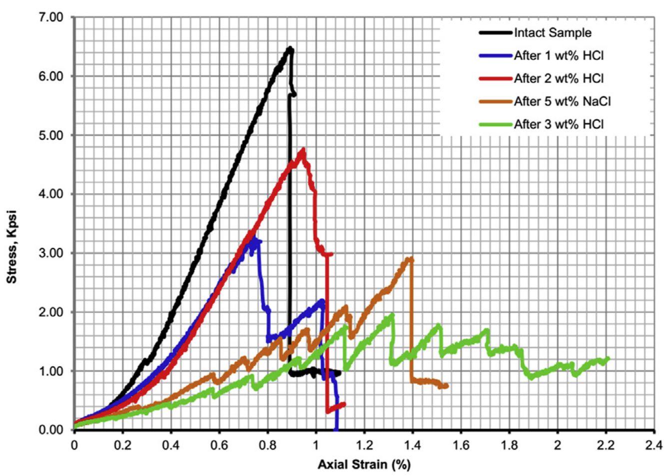
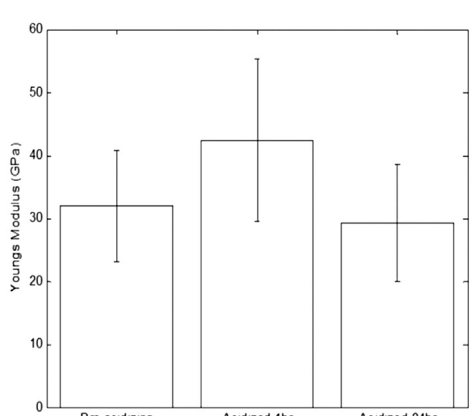


Fig. 5. Stress–strain data for Eagle Ford samples.

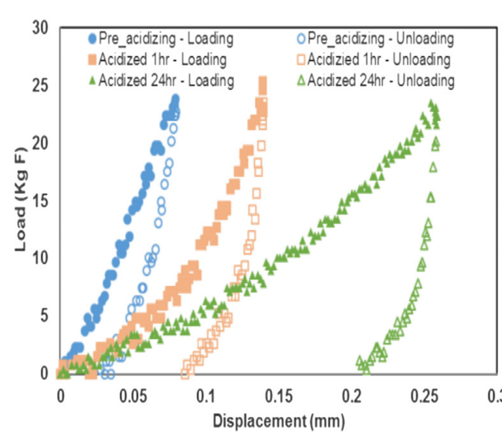
Acid is widely used to reduce the initiation fracture pressure during the hydraulic fracturing. Because acid not only can reduce rock hardness, but also can reduce tortuosity in near-wellbore region. In shale brittle formation, the explosive action of the perforating charges might create several microfractures at each perforation. When fluid is injected into the perforations, these microfractures may be reopened and extended through the medium that allows easy fracture extension. Because of the high Young's modulus, the stress concentrations at the fracture tips are more intense, so these smaller fractures are less likely to link up. However, under the influence of the complex stresses around the wellbore and perforations, fractures can join together at some point, connecting through narrow paths, sometimes with bends, toward a main, large fracture (see Fig. 7). So the treating fluid has to flow from a region containing a large number of small narrow fractures to a region containing a small number of large fractures. In following this path, the fluid has to move through a series of convoluted tortuous, bending and narrow fractures. Certainly, this tortuosity may produce a significant loss in pressure, resulting in a smaller than expected fracture or even worse, a possible early screen-out [40]. Practices have indicated that acid pre-pads can effectively reduce tortuosity.

2.3. Effect of acid on shale fracture conductivity

(1) A preferable dissolution of streaks having higher calcite content is observed at the fracture face. Because calcite and dolomite are the most main two kinds of acid soluble minerals in shale, Calcite (calcium carbonate) reacts with HCl to produce calcium chloride, water and carbon dioxide, while dolomite reacts with HCl to produce calcium chloride, magnesium chloride, water and carbon dioxide. The reaction rate for calcium carbonate is rapid, while the reaction with dolomite is slower [16].
(2) Acid can create surface roughness on shale fracture walls. On core scale, the depth of the surface roughness in a quarter core exposed to acid for 24 h was smaller than 1 mm (see Fig. 8) Channels were observed in Tripathi and Pournik's [33] study, however the depth of the channel was not given. On field scale, the effect of heterogeneity is believed to be even more significant. The presence of geological features such as calcite veins and calcite filled bedding planes is more likely, and these



(a) Young's modulus of a pre-acidizing Bakken sample and the same sample acidized for 1 hour and 24 hours



(b) Typical load-displacement curves with a loading and unloading cycle for a pre-acidizing Bakken, and the same sample acidized for 1 hour and 24 hours

Fig. 6. The difference of Young's modulus between Morsy and Weiwei Wu.

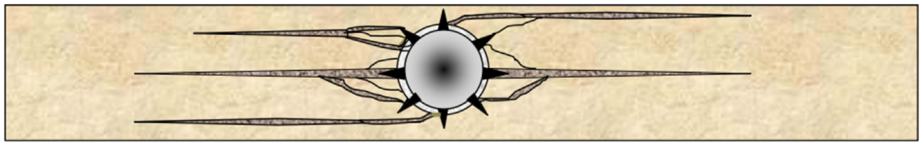


Fig. 7. Multiple fractures and tortuosity.

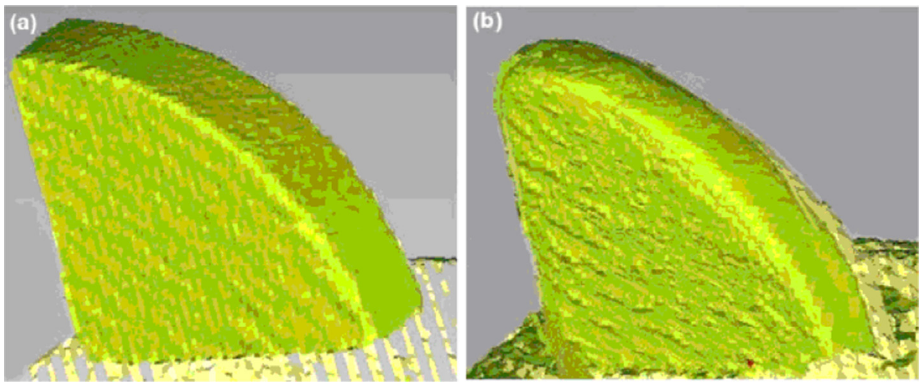


Fig. 8. Comparison of surface profile of pre-acidizing (a) and post-acidizing Bakken (b), in a quarter round with diameter of 1.5 inch. Post-acidizing sample was obtained by pre-acidizing sample exposed to 3 wt% acid for 27 h at ambient temperature. Surface profile was generated by Konica Minolta Vivid 910 3D non-contact digitizing system.

features can easily create non-uniform etching and large scale surface toughness such as channels that can promote the flow of hydrocarbons.

(3) Acid makes the shale fracture conductivity increase. Some experiments (proppant-less fracture, partially propped fracture, propped fracture) show a drop in the fracture conductivity after acidization, but these experiments are at variance with objective reality, because shale is pressurized during acid fracturing job. Therefore, pressurized acid soaking experiment is more compatible with the actual situation, and this experiment result (see Fig. 9) also shows an increase in the fracture conductivity after acidization.

Acidized fracture conductivity was found decrease from around 5–20 md.ft (by around 98%) when the closure stress was increased from 500 psi to 3500 psi [33]. Such measurements probably do not reflect the non-uniform etching that is expected to happen in the field. Since Divyendu Tripathi et al. use the core sample containing carbonate rock up to 60.7%, carbonate rock is dissolved by HCl after acidization, so the fracture face is softened, which leads to embedment of proppant. The experiments are being conducted in 1 inch diameter core. It is likely that the acid will uniformly etch the fracture surface in the lab. Similar situation is not likely to happen in the field where a much larger fracture surface is exposed to the acid and then leads to much more non-uniform etching of the fracture surface. Such a scenario is hard to replicate in the lab.

3. Conclusion and recommendation

(1) Downhole acidic scale removal, matrix acidizing, pad-acid-hydraulic fracturing technology and multistage alternating injection proppant-carrying acid fracturing are main acid treatment technology for shale oil and gas reservoirs stimulation, the stimulation mechanism study mainly concentrates on three aspects, effect of acid on shale matrix, effect of acid on shale mechanical properties, effect of acid on shale fracture conductivity.

(2) Most researchers use rock outcrop to study acid stimulation mechanism for shale oil and gas. Because it may be different for rock outcrop and reservoir rock, so reservoir rock should be used to study acid stimulation mechanism in the future. Moreover, most researchers use small-scale core sample to study shale acid-etched fracture conductivity, which could not be liable to reflect the anisotropy of shale. So a large-sized core sample should be used to study shale acid-etched fracture conductivity.

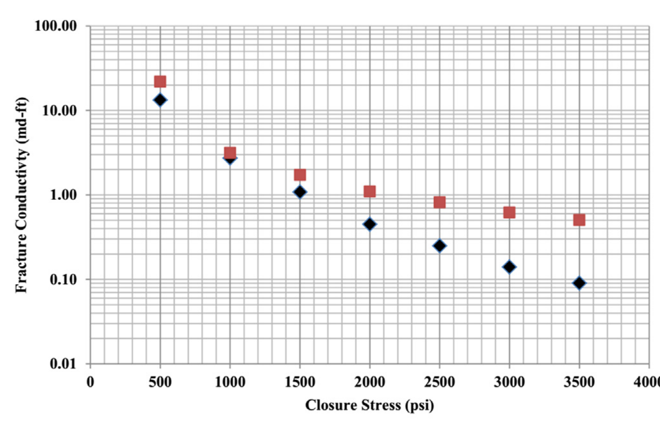


Fig. 9. Effect of pressurized acid soaking in proppant-less fracture.

(3) At present, shale acid treatment technology is mainly applied to shale of high carbonate content. Therefore, in order to avoid excessive acid dissolution on carbonate rock, mild acid strength should be used in this case. And due to shale mineral composition's complexity, each case of shale oil and gas reservoirs should be treated, respectively. In addition, novel acid system should be developed to reduce the acid cost and damage for shale reservoir.
(4) The difference between shale oil and gas reservoir for acid stimulation is a future research direction.

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