



## Review

# New Insight into Enhancing Organic-Rich Shale Gas Recovery: Shut-in Performance Increased through Oxidative Fluids

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**Abstract:** Oxidizing stimulation of organic-rich shale reservoirs, as a supplement of hydraulic fracturing, was proposed to enhance shale gas recovery. Previous publications revealed that the interaction between organic-rich shale and oxidative fluids causes the components' dissolution, which induces lots of pores and microfractures, resulting in rock microfracturing without confined pressure and associated increments of the matrix permeability, and improving unproped fracture conductivity. However, the enhancement of shale gas recovery with oxidative fluids still lacks an implementation clue targeted for specific engineering problems. In recent years, water-rock interaction inducing microfractures indicates a positive effect of retained fracturing fluid on the stimulation after the fracturing operation, which sheds light in the enhancement of shale gas production by shut-in. The objectives of this study are to provide a new perspective whereby the shut-in performance to enhance shale gas recovery could be increased by the injection of oxidative fluids into the formation during the fracturing operation. Firstly, the mechanisms of shut-in performance increased by oxidative dissolution, which illustrate the increment of the density of fracture networks, the improvement of fracture network conductivity, and the promotion of gas desorption and diffusivity, are demonstrated. Then, the feasibility of using oxidative fluids to increase shut-in performance, which follows the geological and engineering characteristics of organic-rich shale reservoirs, is evaluated. Finally, according to the analysis of production performance for two typical types of shale gas wells, in which one is a low gas production and a high fracturing fluid recovery (LGP-HFR) and the other is a high gas production and a low fracturing fluid recovery (HGP-LFR), a shut-in strategy with oxidative fluids to enhance shale gas recovery is developed. This indicates that the injection of oxidative fluids during the fracturing operation may become a promising and cost-effective approach to enhance shale gas recovery.

**Keywords:** organic-rich shale; formation damage; shut-in; oxidation; dissolution; water-rock interaction



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## 1. Introduction

The gas production of shale gas reservoirs is enhanced with the implementation of hydraulic fracturing technology. Unfortunately, the rapid decline of gas production in the early stage followed by a low gas recovery is still widespread [1–3]. Normally, there are three major viewpoints to explain this phenomenon according to previous studies. For the first one, as it is known, shale gas in the matrix pore usually experiences multi-scale transport processes which involve desorption, diffusion, and percolation before it enters into the wellbore [4–7]. Especially, the desorption and diffusion efficiency of the adsorbed gas or free gas in nanopores is so low that the gas transport capacity is extremely hindered [8,9]. Hydraulic fracturing, which creates a complex fracture network, is beneficial to improve the gas flow capacity in the shale reservoirs. However, it still fails to effectively

improve the capacity of gas desorption and diffusion in the matrix nanopores, thus resulting in the gas supply capacity from the matrix being far lower than the gas transport capacity of the fracture network [10–14]. Secondly, hydraulic fracturing creates a complex fracture system consisting of artificial fractures (primary fractures) and stress induced and/or reactivated natural fractures (secondary fractures) [15–18]. The width of the secondary fractures, ranging from  $10^{-1}$  to  $10^{-6}$  cm, is so small that the most commonly used proppants, such as 40–70-mesh or 100-mesh sand, cannot enter efficiently [16–21]. These unpropped fractures, which have a larger contact area with the matrix than the primary fractures, play significant roles in shortening the distance of shale gas transport from the matrix to the primary fracture [22–24]. Regrettably, the width or conductivity of the unpropped fractures are usually decreased due to the stress dependence along with the gas production and fracturing fluid filtration or flowback in these fractures [25–28]. Thirdly, over ten thousand cubics of fracturing fluids is pumped into a shale gas well to create the required fracture network for production. Nevertheless, over 70% of fracturing fluids are retained in the formation after the flowback operations, which induces some formation damage such as aqueous phase trapping (APT) or clay swelling, impairing the performance of the fracturing stimulation [29–32]. Reducing the amount of the retained fluid and shortening the water–rock interaction time through fast flowback operations after hydraulic fracturing can facilitate the reduction of these formation damages [33–38]. Nowadays, considering the irreplaceable hydraulic fracturing operation and followed inevitable fracturing fluid retention, how to treat the aforementioned problems economically and environmentally and further enhance shale gas recovery has become a research hotspot.

Field production data of shale gas wells showed that shut-in after the fracturing operations significantly increases gas production and reduces water production in the production process of the shale gas well, in which the gas production will increase with shut-in time [39–43]. Firstly, the increment of gas production is attributed to the matrix stimulation induced by water–rock interaction after the fracturing operation, which is regarded as a beneficial supplement to the stimulation by hydraulic fracturing [43–49]. Secondly, imbibition and redistribution of the retained fracturing fluid, which contributes to the removal of APT in the fracture network, has an important effect on the increment of early gas production [50]. Specifically, the newly generated pores and fractures due to the water–rock interaction promote the redistribution of fracturing fluid retained in the fracture network [51]. Moreover, the displacement of absorbed methane molecules with water molecules occurs during the fluid imbibition and redistribution [52,53], which plays an important role in increasing gas production and slowing the decline rate of the gas production [2,54–56]. Thirdly, the width of the unpropped fracture will gradually reduce, even to a close with the production time, resulting in a production decline of the shale gas well [23,57,58]. Interestingly, the fluids retained in these fractures might function as a proppant to prevent them from closing if the gas can bubble or channel to the horizontal borehole through the water phase in the fractures [59]. Moreover, to avoid or mitigate formation damage due to the flowback operation [38], while alleviating the environmental contaminants associated with flowback wastewater, a promising strategy of sequestering the fracturing fluid permanently in a shale formation was proposed by Yang et al., 2019 [60]. Similarly, You et al. (2021) proposed the concept, feasibility, and significance of the zero flowback rate (ZFR) of hydraulic fracturing fluid in shale gas reservoirs and illustrated the effect of ZFR on formation damage control, the matrix permeability enhancement, cost saving, and environmental maintenance [61]. To summarize, a proper combination of fracturing fluid retention and a design of the shut-in strategy may have a good potential for and impact on enhancing the production of shale gas wells.

Fracturing fluid retention in shale gas wells has pros and cons on gas production. Only when the profit exceeds the loss can the shut-in performance occur. To strengthen the benefit effects while lessening the negative effects, You et al., 2017 mentioned that the fracturing fluid retention can create a favorable condition for formation stimulation and proposed and demonstrated a new idea to enhance shale gas recovery through oxidation-

induced rock bursts, which simply requires the injection of oxidation agents into the shale gas reservoirs [14]. Since the idea of applying oxidizing stimulation into organic-rich shale gas reservoirs was proposed, it has attracted wide attention and continuous research. Previous publications revealed that the interaction of shale and oxidative fluids can cause the dissolution of organic matter (OM) and rock minerals, inducing pores and microfractures, rock microfracturing without confined pressure, and an associated increment of the matrix permeability and unpropped fracture conductivity [13,62–70]. However, the application of oxidative fluids for the enhancement of shale gas recovery still lacks a comprehensive implementation clue targeted for specific engineering problems. The objectives of this study are to provide a new perspective whereby the shut-in operation to enhance shale gas recovery could be enhanced through oxidative fluids during the fracturing operation. Initially, the mechanisms of shut-in efficiency increased by oxidative dissolution are demonstrated. Then, the feasibility of using oxidative fluids to increase shut-in performance is evaluated. Finally, a shut-in implementation clue with the application of oxidative fluids to enhance shale gas recovery is developed.

## 2. Mechanism of Shut-In Performance Increased by Oxidative Dissolution

The previous section indicated that shut-in performance depends on water–rock interaction-induced microfractures, the auto-release of APT in the fracture network, and the output of adsorbed gas. This section will reveal how the oxidative dissolution plays a role in all these aspects that contribute to increasing shut-in performance. Therefore, the positive effect of oxidative dissolution on the increment of the density of fracture networks, the improvement of fracture network conductivity, and the promotion of gas desorption and diffusivity are analyzed.

### 2.1. Increment of the Density of Fracture Networks

Shale matrix is crushed as much as possible to improve the density of fracture networks, which contributes to a high gas production and consequent high gas recovery [13,14]. Currently, in order to improve the density of the fracture network, a small amount of acidic fluids usually was pumped into the formation prior to the fracturing operation, which can decrease the fracture pressure of the rock through the dissolution of carbonate minerals or other rock cements [71–75]. This has proven to be a good process according to the reported field data of shale gas wells from either the ones in north America, such as Bakken and Engle Ford formation, or the ones from the Sichuan basin in China [76,77]. Fracture pressure, as an intrinsic property of rock formation, is controlled by the mineral composition, the mineral content, and the layout of the rock. For example, in open hole completion conditions, the calculation formulas of fracture pressure which induces vertical or horizontal fractures are indicated as Equations (1)–(3). This shows that physical properties of the rock such as porosity ( $\phi_c$  and  $\phi$ ) and mechanical parameters such as the Poisson ratio ( $\nu$ ) and uniaxial tensile strength ( $\sigma_f$ ) dominate the reduction of the fracture pressure [78]. Tugrul et al. (2004) experimentally measured the porosity of the rock and its uniaxial tensile strength for sandstone, limestone, basalt, and granodiorite before and after weathering oxidation, which showed that the value of uniaxial tensile strength displays an exponential decline with the increasing total porosity [79]. On a macroscale, acidic fluids impair the strength of cementation by reacting with the rock cements, which reduces the cohesive force and internal friction angle of the rock. On the other hand, on a microscale, the generation of macroscopic fractures usually is the result of a process whereby many microfractures in the rock gradually open, expand, and connect due to loading stress and then accumulate to a certain extent. Acidic dissolution may have a positive influence in this process. On one side, acidic dissolution-induced pores and microfractures contribute to increase the porosity of a rock, which causes the chemical damage of the rock, resulting in the reduction of the uniaxial tensile strength [80,81]. Equation (4) shows a chemical damage variable dependent on porosity ( $D_\phi$ ), which reflects the chemical damage due to the interaction between acidic fluids and shale [82,83]. On the other hand, Lin et al. (2016)

found that elastic modulus and uniaxial compressive strength are negatively exponentially correlated with the chemical damage variables for shale samples treated by acidic fluids, respectively [84]. Thus, there is another chemical damage variable, named as  $D_E$ , which is dependent on the elastic modulus (Equation (5)). Further, the constitutive relation of the rock chemical damage with the elastic modulus as a damage variable is shown as Equation (6) [85].

$$P_{bv} = \frac{3\sigma_h - \sigma_H + \sigma_f - 2\eta P_o}{1 + \phi_c - 2\eta} \quad (1)$$

$$P_{bh} = \frac{P_{ob} + \sigma_f - 2\eta P_o}{\phi_c - 2\eta} \quad (2)$$

$$\eta = \frac{\phi(1 - 2\nu)}{2(-2\nu)} \quad (3)$$

$$D_\phi = \frac{\phi_w - \phi_o}{1 - \phi_o} \quad (4)$$

$$D_E = 1 - \frac{E_w}{E_o} \quad (5)$$

$$\sigma_w = E_o(1 - D_E)\varepsilon_w \quad (6)$$

where,

$P_{bv}$  stands for broken pressure for vertical fracture, MPa

$P_{bh}$  stands for broken pressure for horizontal fracture, MPa

$\sigma_h$  stands for minimum horizontal principal stress, MPa

$\sigma_H$  stands for maximum horizontal principal stress, MPa

$\sigma_f$  stands for uniaxial tensile strength, MPa

$P_{ob}$  stands for overlying formation pressure, MPa

$P_o$  stands for fluid pressure in pores, MPa

$\phi_c$  stands for porosity of rock contact point

$\phi$  stands for porosity

$\nu$  stands for Poisson ratio

$D_\phi$  stands for chemical damage variable depended on porosity

$D_E$  stands for chemical damage variables depended on elastic modulus

$\phi_w$  stands for porosity after chemical damage

$\phi_o$  stands for porosity before chemical damage

$E_w$  stands for elastic modulus after chemical damage, GPa

$E_o$  stands for elastic modulus before chemical damage, GPa

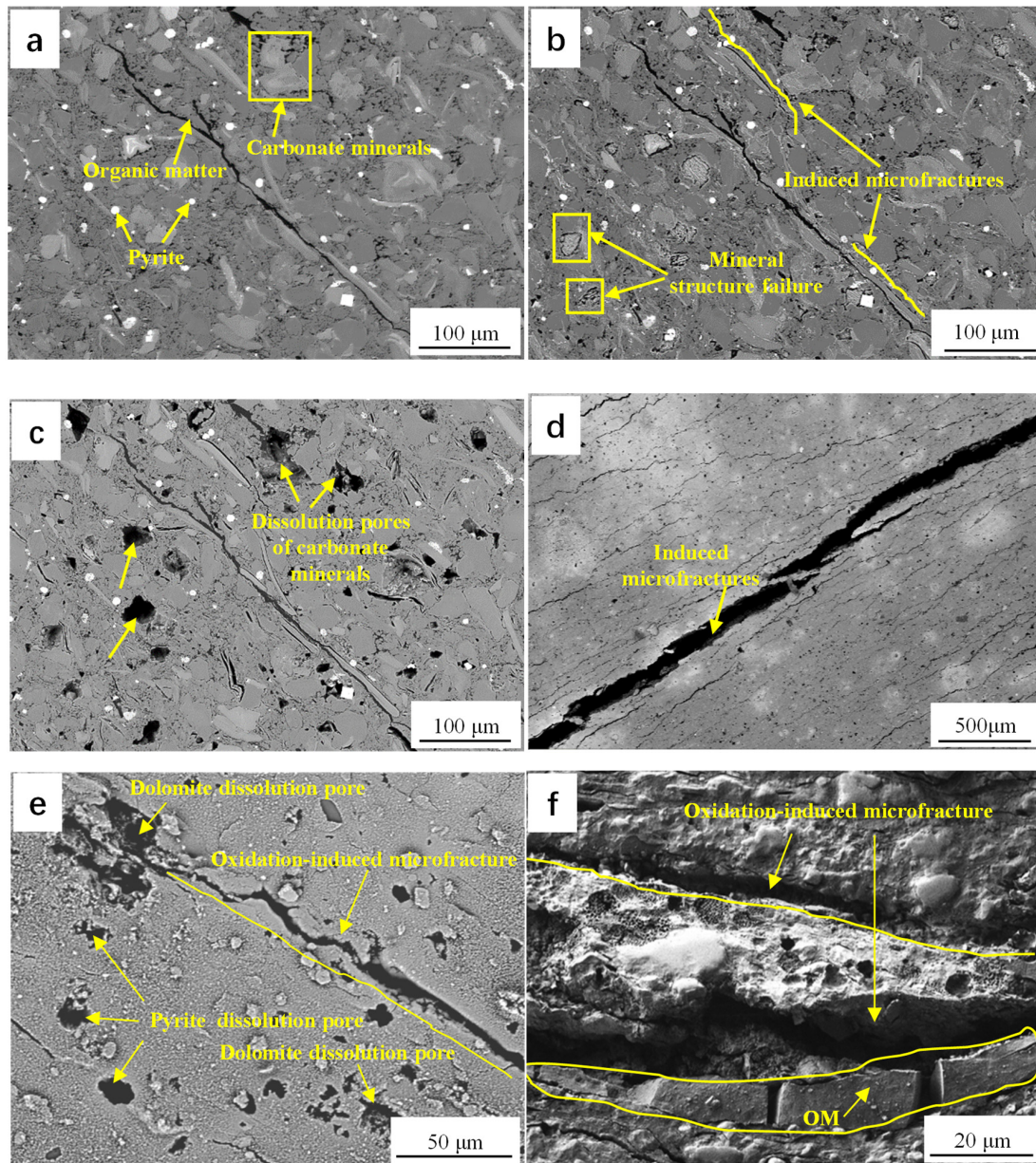
$\sigma_w$  stands for stress after chemical damage, MPa

$\varepsilon_w$  stands for strain after chemical damage

Similarly, oxidative fluids may play the same role as acidic fluids in decreasing the fracture pressure of rocks in organic-rich shale reservoirs. Previous publications have revealed a change in components including OM, pyrite, and carbonated minerals during the shale and oxidative fluids reaction [13]. The dissolution of carbonate minerals may be caused by the hydrogen ion ( $H^+$ ) in the oxidant or the  $H^+$  generated in the process of the oxidation reaction [13,69]. Especially, an acidic oxidant such as hydrogen peroxide ( $H_2O_2$ ) co-exists with acidic fluids such as hydrogen chloride (HCl). This means that oxidative fluids containing acidic agents may have a stronger potential for the dissolution of shale rocks. Meanwhile, oxidative dissolution-induced pores and microfractures of the organic-rich shale sample from the Sichuan basin have been reported [13,69]. This study, repeated previously, reported methods to compare the structure change of organic-rich shale samples after the treatment of distilled water, acidic fluids, or oxidative fluids, respectively. An observation of Figure 1 indicates that oxidative fluids not only induce a greater amount of microfractures compared with distilled water but also result in more dissolution pores and microfractures compared with acidic fluids. You et al. (2018) reported that a higher concentration of oxidative fluids results in a greater increment of the porosity of shale plugs.



They showed that the porosity of shale plugs treated by 15% wt.  $H_2O_2$  for 72 h have an increment of 36.4% compared to that of the untreated sample and an increment of 49.3% compared to that of the sample treated with distilled water [70]. Oxidative dissolution thereby shows a great potential for increasing shale porosity and hence decreasing fracture pressure, according to Equations (1)–(4).



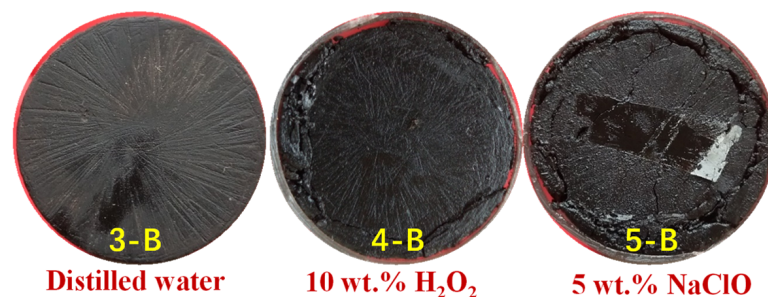
**Figure 1.** Field-emission scanning electron microscopy (FESEM) images of organic-rich shale sample: (a–c) In-situ observation of shale sample from the Longmaxi formation in the Sichuan basin, in which the untreated sample (a) is exposed to distilled water (b) and acidic fluids (c) for 120 h, respectively (adapted from Chen et al. (2018) [86]). (d,e) Shale sample from the Longmaxi formation in the Sichuan basin is exposed to oxidative fluids (15% wt.  $H_2O_2$ ) for 240 h (adapted from Chen et al. (2017) [13]). (f) Source rock shale samples from the Middle East are exposed to oxidative fluids containing 0.0087 M ammonium persulfate, 0.013 M sodium bromate, and 0.27 M potassium chloride in 10 mL of water for 20 h at 100 °C (adapted from Hull et al. (2019) [66]). Note that acidic fluids only caused the dissolution of the carbonate minerals, while oxidative fluids caused the dissolution of pyrite and OM besides the carbonate minerals. Oxidative dissolution induced lots of pores and microfractures.

In order to further explore and prove the above viewpoint for oxidative fluids, organic-rich shale plugs, which are treated by distilled water or oxidative fluids for 10 days, respectively, were used to carry out the triaxial compression test. These plugs, obtained from the Longmaxi formation in the Sichuan basin, which have been reported in our previous publications [13,68–70], were drilled cores, paralleling the lamination at the same location of a big shale block sample. Table 1 shows that the compressive strength of all the plugs after the treatment of oxidative fluids displays a reduction of varying degrees, contrasting to that of plugs treated by distilled water. Meanwhile, observations of all the plugs experienced in the triaxial compression test find that oxidative fluids induce a more severe fracturing of the rock with the same stress loading (Figure 2). Liu and Sheng (2019) found that axial stress plays an important role in promoting water–rock interaction, inducing shale rock fracturing, in which the macrofracture extends by linking the microfractures due to the axial stress [87]. This indicates that oxidative fluids contribute to accelerating and aggravating the generation and propagation of tensile fractures with the axial stress loading.

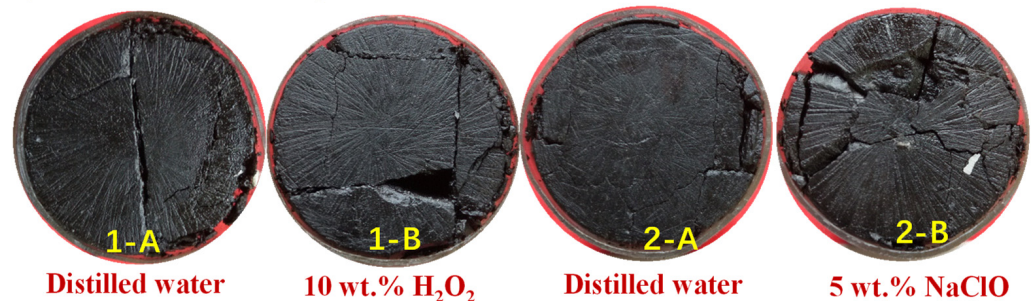
(Confined stress = 10 MPa)



(Confined stress = 20 MPa)



(Confined stress = 30 MPa)



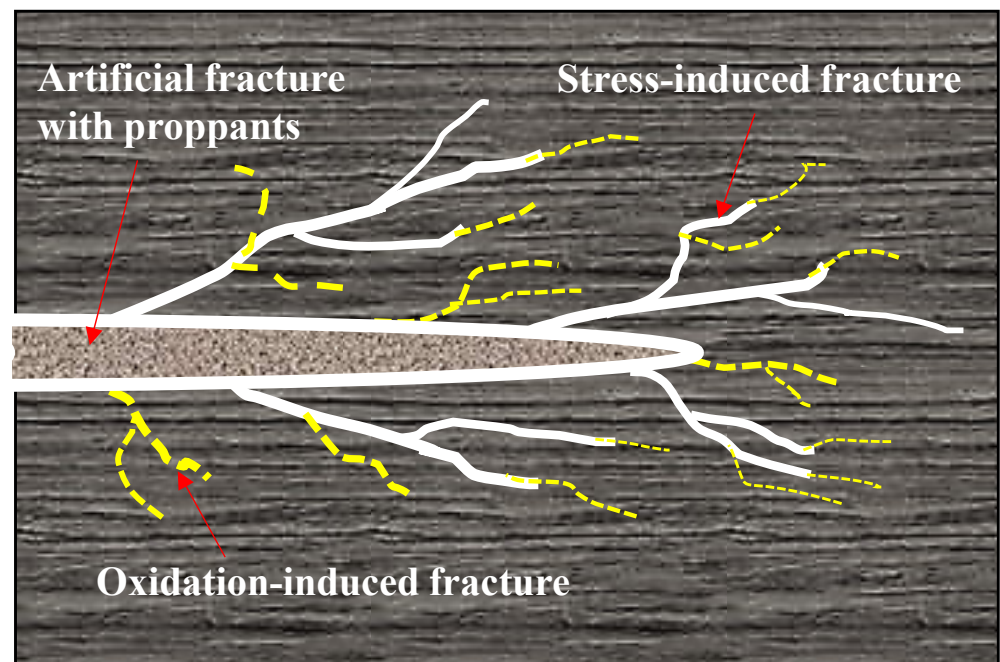
**Figure 2.** Observation of shale rock plugs that experienced the rock triaxial compression test. All shale samples treated by oxidative fluids generated more severe fracturing compared to those treated by distilled water under the same stress loading. Note that sample A and B means these two samples obtained and cut from the same long shale plug samples.



**Table 1.** Triaxial compression test of treated shale samples. Note that no naked visible microfractures are observed from the surface of all the shale samples. Each group of samples (labelled as A and B), obtained from the same long plug which was cut into half to ensure that two samples were being compared, have similar physical properties.

Sample ID	Diameter, mm	Length, mm	Experimental Fluids	Confined Pressure, MPa	Elastic Modulus, MPa	Compressive Strength, MPa	Poisson Ratio
1-A	25	48.5	Distilled water	30	28,289	267	0.37
1-B	25	48.0	10 wt.% H <sub>2</sub> O <sub>2</sub>	30	19,756	213	0.35
2-A	25	49.0	Distilled water	30	26,627	238	0.30
2-B	25	47.0	5 wt.% NaClO	30	26,246	197	0.22
3-A	25	49.0	Distilled water	10	27,660	278	0.15
3-B	25	47.5		20	26,498	294	0.27
4-A	25	49.0	10 wt.% H <sub>2</sub> O <sub>2</sub>	10	28,372	266	0.24
4-B	25	48.0		20	25,633	267	0.28
5-A	25	48.5	5 wt.% NaClO	10	27,353	275	0.19
5-B	25	48.5		20	22,424	192	0.10

In the field, the pressure in the fracture network's (fluid pressure) post-fracturing operations will reduce with the filtration of the fracturing fluid into the matrix gradually. When the fluid pressure in the fracture reaches the minimum horizontal principal stress of the formation, the potential effect of shut-in on fracture initiation and propagation will disappear completely [88]. In this period, if the fracturing fluid contains an oxidative agent that is designed to decrease the fracture pressure of shale rock, current shut-in performance focusing on the increment of the density of fracture networks is based on the effect of oxidative dissolution (Figure 3).



**Figure 3.** Schematic diagram of the density of fracture networks increased by shut-in with the support of oxidative dissolution in organic-rich shale gas reservoirs.

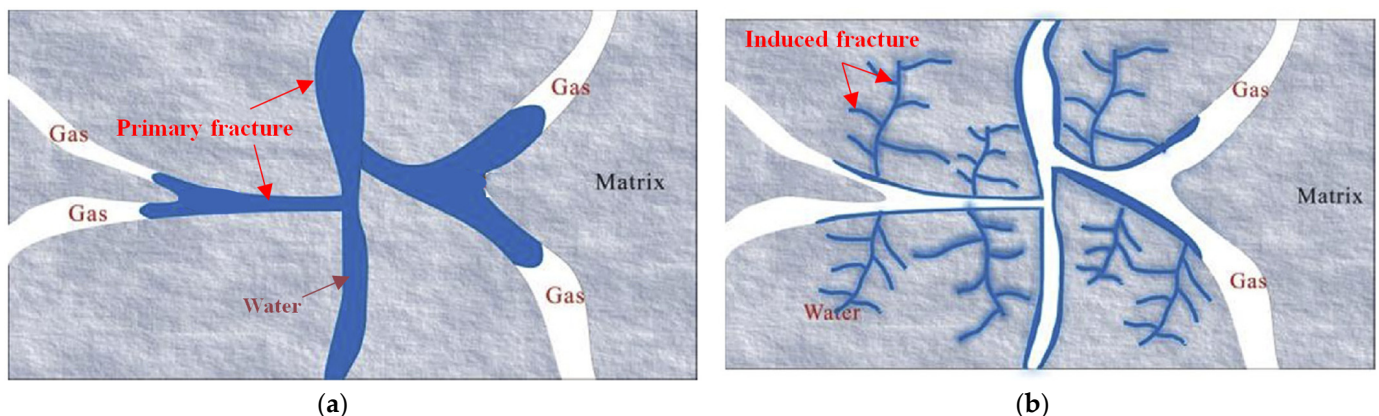
## 2.2. Improvement of Fracture Network Conductivity

A higher density of fracture networks leads to a more efficient gas transport from the matrix to the wellbore, which is a sign of obtaining higher shale gas production. Unfortunately, fracturing fluid retained in the fracture usually causes APT, which reduces the fractures' conductivity or relative permeability, which results in an artificial reduction of the EFV. Moreover, Xu et al. 2017 reported that pressure depletion and fracture closure during the flowback operation in early stages might lead to over 30% of the effective fracture volume (EFV) loss, which is regarded as one of the important factors for a rapid

decline in early gas production [26]. The conductivity of unpropped fractures could reduce with the production time due to stress dependence, resulting in a further reduction of EFV [19,27,28]. In the following subchapter the mechanism of the oxidative dissolution solving two issues mentioned above is explored.

### 2.2.1. Auto Removal of APT in Fracture

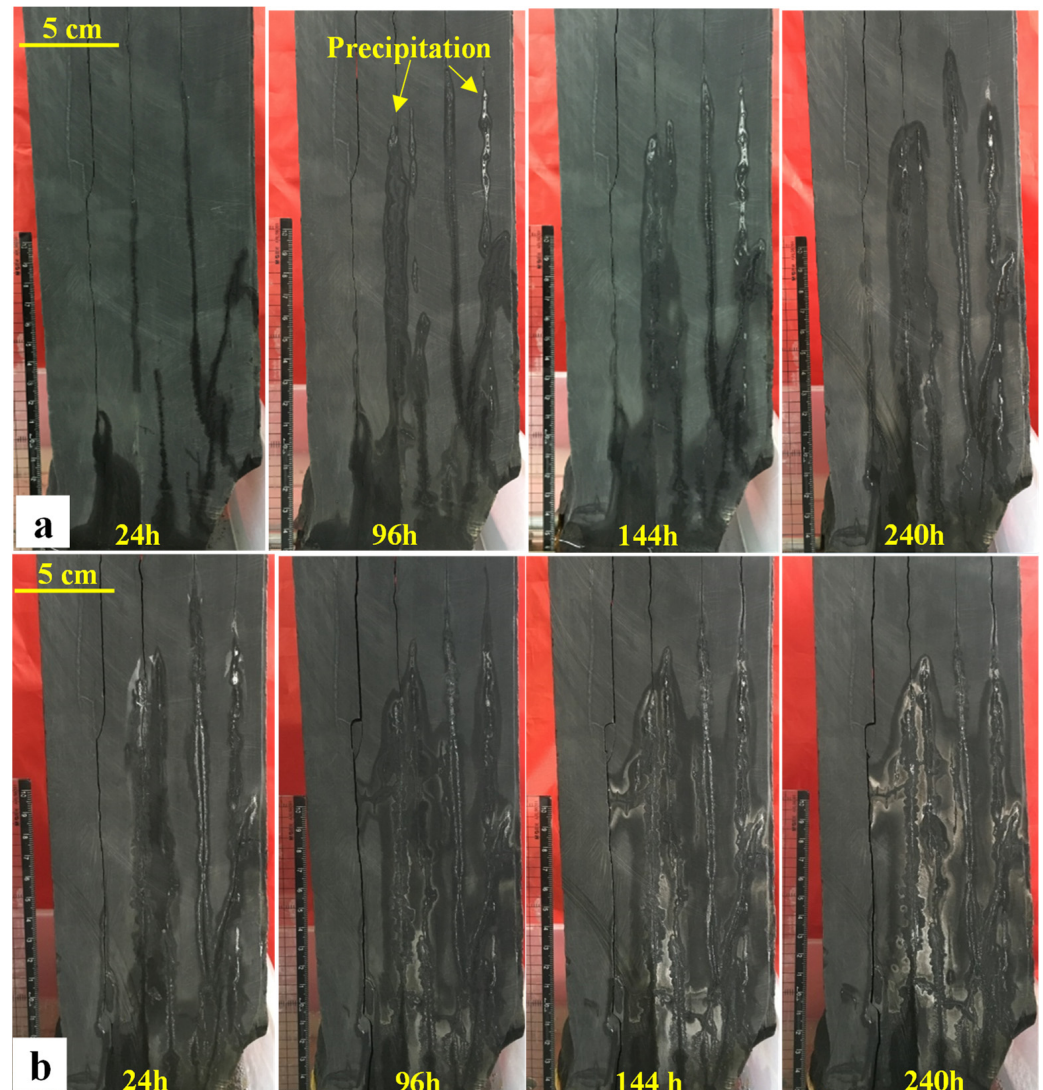
In tight sand gas reservoirs, a high flowback efficiency of the fracturing fluid is a guarantee to gain high gas production. Unfortunately, this approach does not work in shale gas reservoirs due to the extremely low fluid recovery, owing to the unique geological characteristics, such as strong water imbibition and retention capacity [35–37]. Shale matrix pores, particularly the micropores, have a preference for water imbibition, according to the experiment of spontaneous imbibition with displacing gas [89]. In the process of water imbibition and redistribution, these pores can uptake water from other pores, especially from the main flowing channels [90]. However, fracturing fluid imbibes and diffuses through the shale matrix, and the associated clay swelling induces fractures in this process, which are the primary auto-removal mechanism of APT with shut-in in shale reservoirs [30,51,90,91]. Specifically, the fracture in shale rocks, which plays a significant role in the increment of volume imbibed and the improvement of the imbibition rate of fracturing fluids, promotes the redistribution of the fluid [39]. This implies that the fluid retained in the fracture network imbibes and diffuses into a deeper matrix, then accompanies the generation of induced fractures. This mechanism contributes to the reduction of water saturation in the primary fracture, thus improving the primary fracture conductivity, as shown in Figure 4.



**Figure 4.** Water block unlocking effect for water diffusion into a deeper matrix (a), accompanying the generation of induced fractures (b) (adapted from Shen et al. (2016) [51]).

For the generation of induced fractures, the last section explored that oxidative dissolution induces fracture initiation and propagation with the help of fluid pressure in the fracture during the shut-in period. Moreover, Figures 1 and 2 show that oxidative fluids induce more microfractures compared with distilled water or conventional fracturing fluid at a micro-scale. Similar results were obtained in the black shale plugs after their exposure to distilled water and oxidative fluids, respectively [13]. This result indicates that oxidative fluids spreading to the shale matrix have a great potential on the initiation and growth of fractures. You et al. (2018) found that the curve of the imbibed volume vs. time occurs a step increase with a sudden generation of fractures during the imbibition of high concentration  $H_2O_2$  in the organic-rich shale. For the low concentration of  $H_2O_2$ , an increment of the imbibition potential owes to the oxidation-induced pore and fractures was revealed, according to the typical Handy model [72]. Figure 5 shows that the generation of induced fractures is beneficial to the water diffusion and redistribution from the primary fracture to induced fractures. An observation of the shale samples can prove that oxidative dissolution enhances the scope of water distribution in the same imbibition time, which

is judged according to the footprint of the fluid and the distribution of the precipitations. These precipitations are recognized as soluble salts which evolve from the fracture, indirectly indicating that the generation of microfractures during the imbibition contributes to the water redistribution. Moreover, the increment of the fracture surface area with the generation of induced fractures also helps to infer that fluid imbibes and diffuses into the matrix near the fractures [92,93]. As a result, the APT in the primary fractures can induce auto-removal further with the effect of oxidative dissolution.



**Figure 5.** Changes of shale samples' surfaces during the imbibition of distilled water (a) and oxidizing solution (b). Note that these samples are from the same big block shale sample, which is used for the imbibition of distilled water first and then dried up at a low temperature for 48 h, before the resumption of imbibition of the oxidative fluids (adapted from Cheng et al. (2020) [92]).

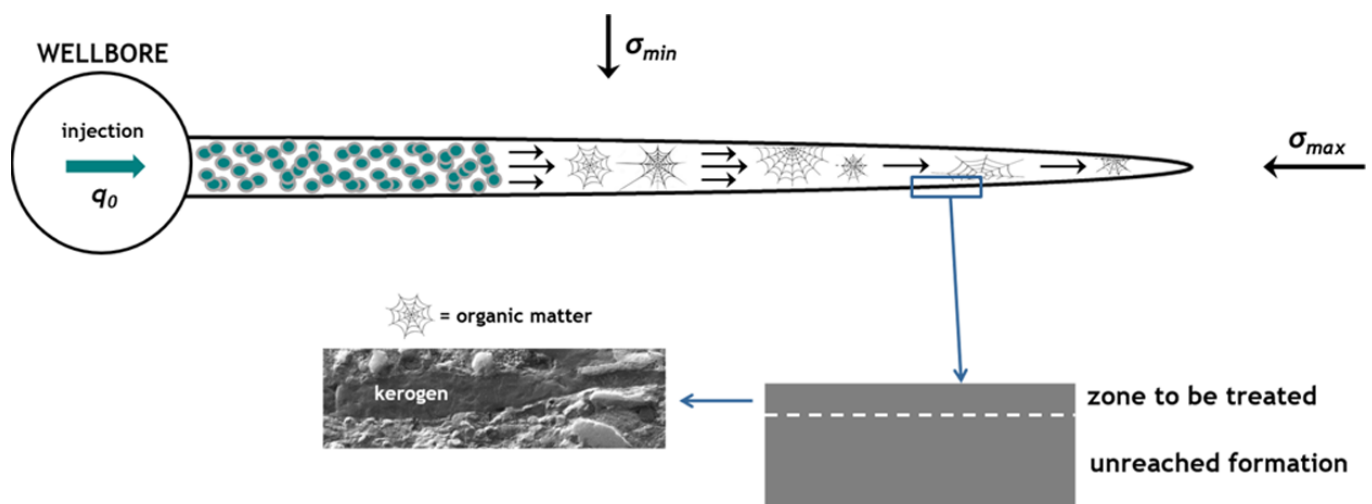
For the water uptake of the shale matrix, Cheng et al. (2020) found that some shale samples, which cannot be induced fractures with the treatment of oxidative fluids, still displayed an increment of imbibed volume of distilled water by 11.6–15.3% and an increment of the imbibition potential by 2.32–8.26% [92]. Furthermore, the oxidation-induced pore and fracture (Figure 1), which increase the occupancy space of water, and the adsorption effect of clay minerals on the water cannot be ignored [57]. Yang et al. (2016) reported that the imbibition capacity of shale rocks is positively correlated with the total clay content, especially the content of illite/smectite mixed-layer clay and smectite [94]. Clay minerals with a large surface determine an enrichment of up to 85% of



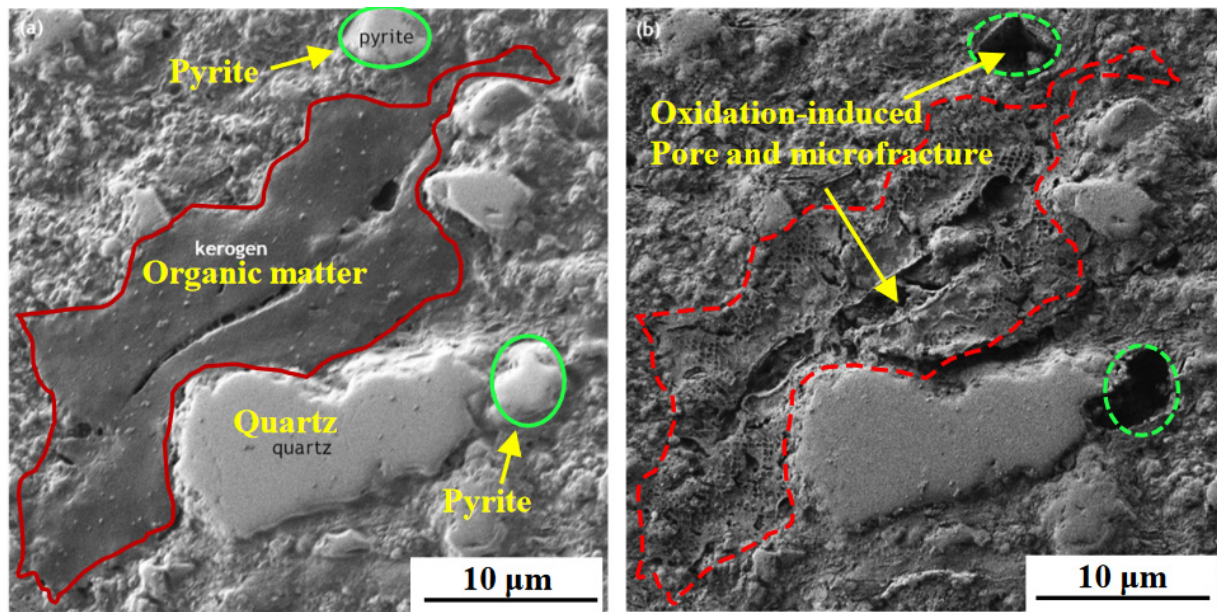
the OM in shale rocks, which generally exists in the form of the mineral-combined OM (known as organo-clay complexes) [95–97]. Clay minerals with a large surface enrich the OM (generally exist in the form of the mineral-combined OM) in shale rocks by up to 85%. The oxidative dissolution of OM can destroy the organo-clay complexes, which increases the contact area of the clay and water and then changes the water distribution in the matrix, reducing the free water amount in the matrix pore [98]. This may play a role in the removal of the APT, which is not only in the fracture but also in the matrix pores. Moreover, improvement of water uptake for the matrix will also be helpful for the removal of APT in the fracture.

### 2.2.2. Non-Uniform Surface-Etching Profile

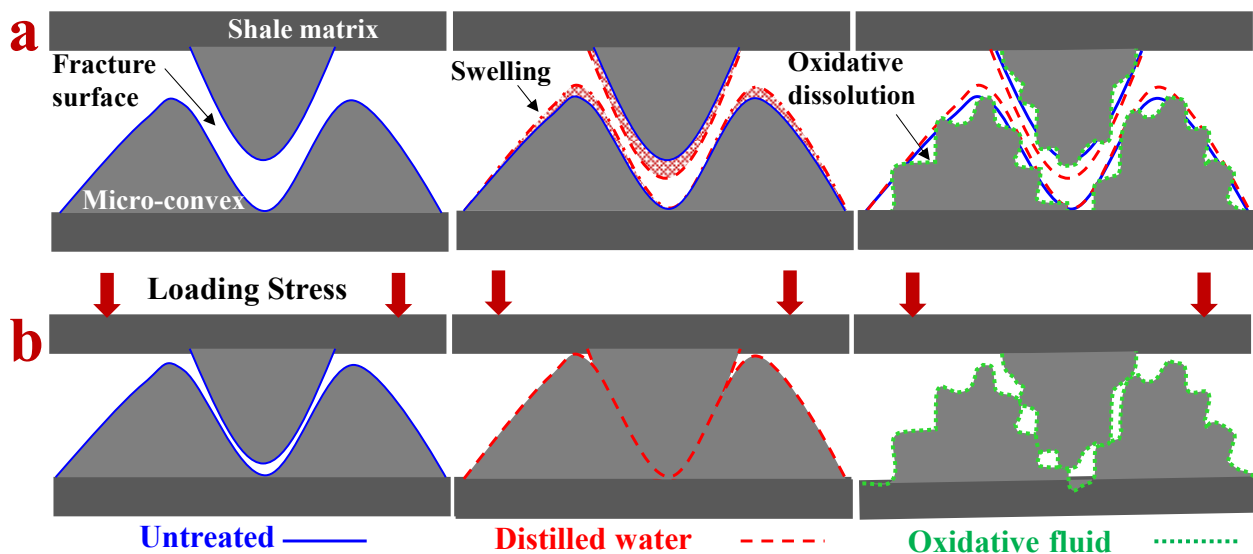
Acid treatment, as a beneficial stimulation in carbonate-rich shale reservoirs due to an increase in the porosity and favorable etching pattern, plays a significant role in maintaining the proppant-less fractures that are open and conductive even in the closure stress regime [19,99]. Similarly, Hull et al. (2019) [66] postulated that the dissolution of OM by oxidative fluids has a huge potential for improving fracture face conductivity (permeability), minimizing proppant embedment, and maintaining the long-term productivity of shale gas wells (Figure 6). Cheng et al. (2021) revealed the effect of oxidative dissolution on increasing the width of unpropped fractures by calculation, according to the dissolution volume of shale components [71]. Figure 7 shows that oxidative dissolution induces a non-uniform surface-etching profile on the fracture face. Meanwhile, the dissolution induces microfracture networks apart from dissolution pores (Figure 1). These two characteristics contribute to the improvement of channels and the formation of surface asperities of the fracture, allowing the maintenance of a relatively better fracture conductivity during the stress loading (Figure 8). Cheng et al. (2021) reported that oxidative dissolution of shale components can increase the fracture width while reducing the stress dependence of unpropped fractures, contrasting with the adverse effects of distilled water [68]. Currently water-based fracturing fluids, which enhance the stress dependence of fracture permeability, usually reduce the conductivities of unpropped fractures on shale [32]. Consequently, oxidative dissolution could be a promising technique to maintain and improve the conductivity of unpropped fractures.



**Figure 6.** Schematic of a hydraulic fracturing fluid being injected into an unconventional organic-rich shale. (Reprinted with permission from [68]. Copyright 2017 Energy and Fuels.)



**Figure 7.** In-situ scanning electron microscopy (SEM) observation of organic-rich shale before (a) and after (b) the treatment of oxidative fluids. This clearly indicates the dissolution of the OM and pyrite. However, other components, such as quartz, are stable (adapted from Hull et al. (2019) [66]).

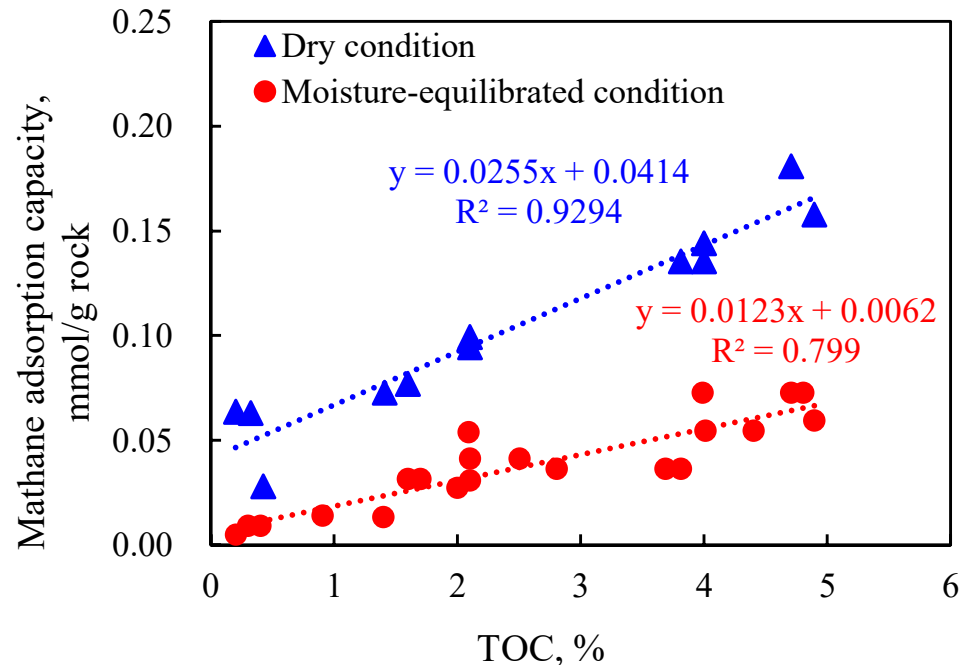


**Figure 8.** Schematic diagram of the surface change of a shale fracture before (a) and after treatment (b) and its closure with the stress loading. Swelling of the clay minerals and fracture surface strength softening triggered by distilled water results in an enhancement of stress dependence [100]. However, oxidative fluids lead to the non-uniform surface-etching profile and induces pore and microfractures, which has a positive effect on resisting fracture closure under effective stress loading.

### 2.3. Promotion of Gas Desorption and Diffusivity in the Matrix

Shale adsorbed gas, which is adsorbed in nano-scale organic and inorganic pores within the matrix, can account for 20~85% of the total gas [101]. TOC content is the dominative factor that affects the adsorption capacity of organic-rich carbonaceous rocks [102,103]. Figure 9 shows a positive linear correlation whereby the improvement of the adsorption capacity in shale rocks with the TOC content increase [104]. Zhu et al. (2014) reported that shale samples with higher TOC content have greater specific surface areas, which can provide more adsorption sites, thus increasing the adsorption capacity [105]. Xiong et al. (2017) found that the ad-

sorption capacity decreases in the following order organic pore > clay mineral pore > quartz pore, and the isosteric heat adsorption of methane decreases upon increasing the pore diameter or decreasing the O/C ratio of OM [106]. Moreover, the adsorption capacity under the same pore diameter decreases with the decreasing O/C ratio [107].

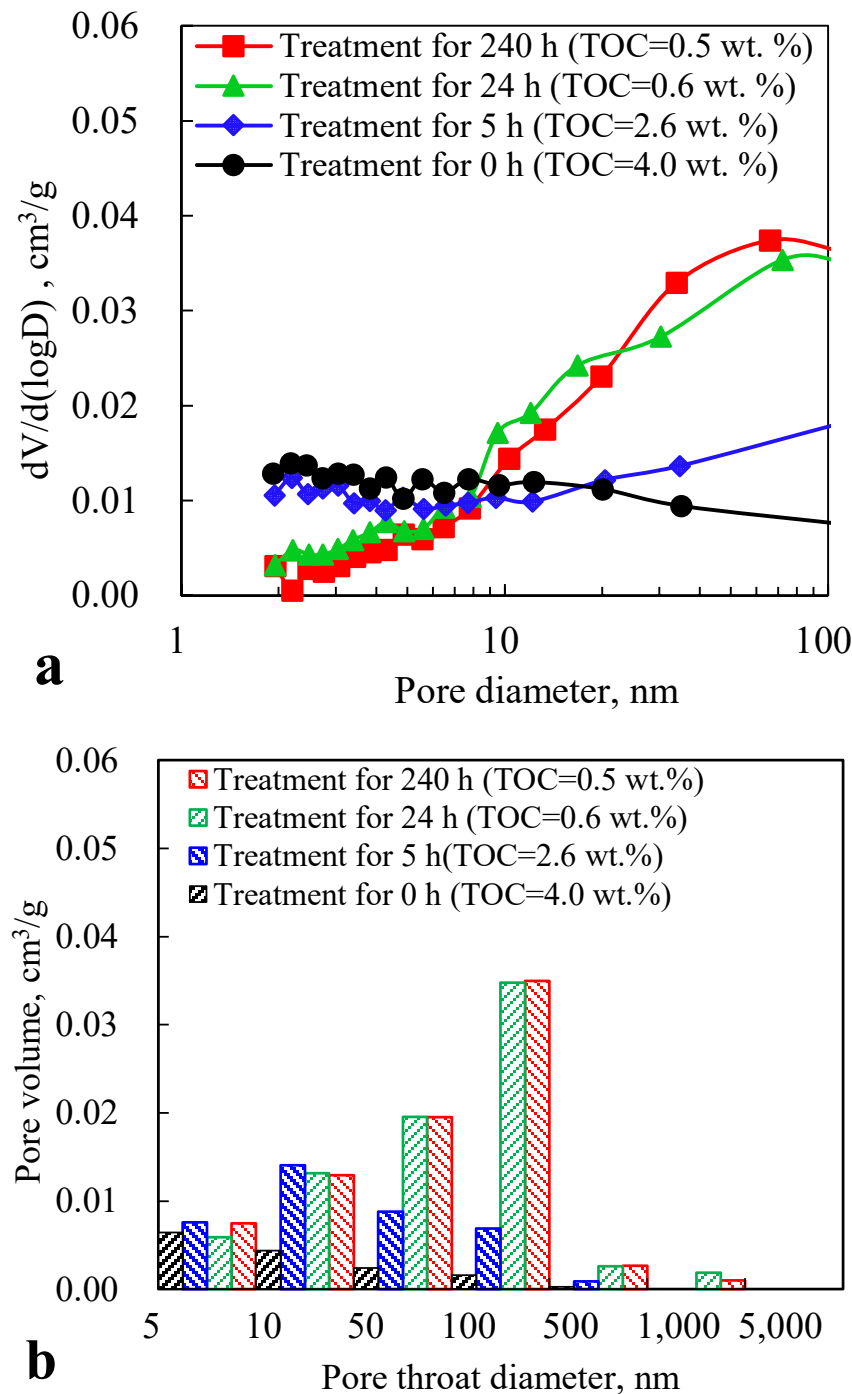


**Figure 9.** Methane adsorption capacity of dry and moisture equilibrated shales samples at 30 C and 6 MPa as a function of TOC content (adapted from Ross and Bustin (2009) [104]).

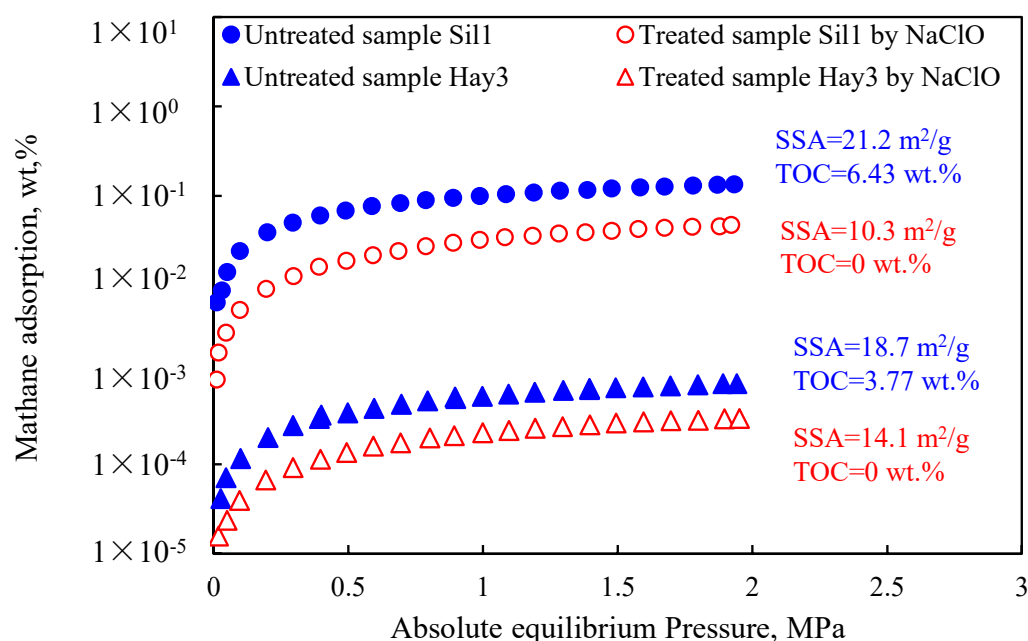
The organic-rich shale–oxidative fluids interaction can effectively realize the removal of the OM. Removal of OM by oxidative fluids according to an in-situ observation of an FESEM image is revealed, as shown in Figure 7. Cheng et al. (2021) found that both  $H_2O_2$  and  $NaClO$  have a good performance for the dissolution of organic-rich shale samples obtained from the Longmaxi, Niutitang, and Yanchang formations in China, respectively [69]. Chen et al. (2017) reported that the removal efficiency of OM by  $H_2O_2$  is up to 87.5% for shale samples from the Longmaxi shale in the Sichuan basin [13]. Moreover, the increment of water adsorbed due to the dissolution of OM contributes to the displacement of methane mainly due to two aspects (Figure 9). One is that the water film bounded on the pore surface changes the interaction characteristics for methane adsorption, and the other is a reduction of the available surface area for methane adsorption because capillary water blocks the pore space [52,53]. As a result, the dissolution of OM by oxidative fluids can have a huge potential on the gas desorption.

Oxidative dissolution of shale components directly causes a change in the pore structure. (Figure 1). Kuila et al. (2014) found a significant reduction of the pore volume network below a diameter of 5 nm due to the removal of OM from thermally mature organic-rich mudrocks [108]. Li et al. (2016) found that after the treatment of  $H_2O_2$ , the increase of the Barrett–Joyner–Halenda (BJH) pore volume and the Dubinin–Astakhov (D-A) micropore volume of an organic-rich shale sample, which was obtained from the Yangchang formation in China, both showed a positive correlation with a decrease of the TOC content [109]. Similarly, for shale samples obtained from the Longmaxi formation in the Sichuan basin, Figure 10a shows a reduction in pore volume for fine mesopores (2–10 nm) and an increase in pore volume at larger pore diameters (10–50 nm) in the process of the removal of OM by  $H_2O_2$ . Meanwhile, Figure 10b shows that the greatest amount of pore volume ranges in the pore-throat diameter from 100 to 500 nm with oxidation for 240 h, according to pore volume distributions calculated from the high-pressure mercury intrusion. Kuila et al. (2014)

insisted that the difference in methane adsorption before and after OM removal roughly correlates to the relative proportion of the nano-pore volume provided by OM and clay textures [108], because the adsorbed gas usually corresponds exclusively to the network of pores with a dimension of <5 nm [101]. Figure 11 shows that shale samples treated by NaClO have a much lower methane adsorption than the natural samples by the methane adsorption experiments with high pressure, which highlights that the presence of OM and associated nanopores play a significant role in controlling methane adsorption.



**Figure 10.** Pore size distribution of shale samples during the oxidative treatment by 15 wt.% H<sub>2</sub>O<sub>2</sub> for 0 h, 5 h, 24 h, and 240 h: (a) pore size distribution of shale samples determined from low-pressure N<sub>2</sub> adsorption; (b) pore volume distributions calculated from high-pressure mercury intrusion (adapted from Chen et al. (2017) [13]).



**Figure 11.** Comparison of supercritical methane adsorption (at 35 °C) between the untreated and NaClO-treated (No OM) shale sample Sil1 and Hay3. This highlights the oxidative dissolution of OM reducing the adsorbed methane capacity of the shale matrix. SSA means specific surface area of the rock pore (adapted from Kuila et al. (2014) [108]).

The enlarging of nano-scale pore size by oxidative dissolution has an influence in accelerating the gas diffusivity in the shale matrix. Yuan et al. (2014) stated that the gas diffusion mechanisms in the shale matrix include Fick diffusion in macropores and Knudsen diffusion in micropores, which shows that gas stored in macropores may be produced quickly and early, while gas stored in the micropores may be produced slowly and late. The diffusion coefficient, an important parameter defining gas diffusion capacity, usually depends on the pore diameter of the shale matrix. Taking Longmaxi shale samples as an example, by the calculation of the diffusion coefficient through the Bidisperse model, the diffusion coefficient in macropores is between  $2.38 \times 10^{-9}$  and  $9.96 \times 10^{-9}$  m<sup>2</sup>/s, and the diffusion coefficient in micropores is between  $1.83 \times 10^{-14}$  and  $3.18 \times 10^{-14}$  m<sup>2</sup>/s [53]. Furthermore, Chen et al. (2018) deeply analyzed the methane diffusion mechanisms in shale and revealed the multiple diffusion behaviors of shale gas [110]. This indicates that gas diffusion contributed by both Knudsen diffusion and surface diffusion will be promoted with the pore diameter enlarged by oxidation dissolution. What is more, gas transport is in forms of transition flow or slip flow because the viscous flow is dominant once the pore diameter is larger than several hundred nanometers. This means that oxidative dissolution can cause a phenomenon whereby partial gas transport in the shale matrix does not go through diffusion and then directly flows into the fracture network.

### 3. Feasibility of Oxidative Fluids Injected to Increase Shut-In Performance

Multi-stage hydraulic fracturing of horizontal wells is successfully applied in the commercial exploration for shale gas reservoirs. A large amount of fracturing fluid is pumped into the formation for creating fracture networks while a small part of the fluid is recovered from flowback operations. Currently, the fracturing operation is irreplaceable for an efficient shale gas recovery, which inevitably results in large amounts of fracturing fluid retention. Shut-in post-fracturing transfers the negative factor of fracturing fluid retention into a positive one by changing the gas's existence state and improving the gas's transport path, which may be effective in increasing shale gas production. Considering that the retained fluid and shale interaction may occur anywhere with respect to the redistribution of the fluid, fully taking advantage of the effects of retained fracturing fluids on post-



fracturing stimulation has aroused wide attention. Currently, considering that conventional fracturing fluid contains 99% fresh water, improvement of fracturing fluid formulas may be an important way to strengthen the water–rock interaction to increase shut-in performance.

Oxidation reaction, which can generate heat and gas and associated pressures, as well as induce chemical dissolution, have been widely utilized in the petroleum industry (Table 2). Oxidative dissolution by  $H_2O_2$ , potassium permanganate ( $KMnO_4$ ), chlorine dioxide ( $ClO_2$ ), sodium hypochlorite ( $NaClO$ ), and ammonium persulfate ( $(NH_4)_2S_2O_8$ ) were investigated for organic-rich rocks, such as coal and shale. This shows a huge potential for the enhancement of primary flows in coal gas formation [111–115]. For organic-rich shale reservoirs, oxidizer breakers such as ammonium persulfate ( $(NH_4)_2S_2O_8$ ) are usually pumped into the formation to realize gel breaking prior to the flowback operation in order to facilitate the fracturing fluid to flow back and reduce formation damages such as APT [116,117]. Therefore, the injection of oxidative fluids into conventional fracturing fluids for increasing shut-in performance post-fracturing is feasible without the extra supplement of equipment and associated costs.

**Table 2.** Commonly used oxidant in petroleum engineering collected from previous publications.

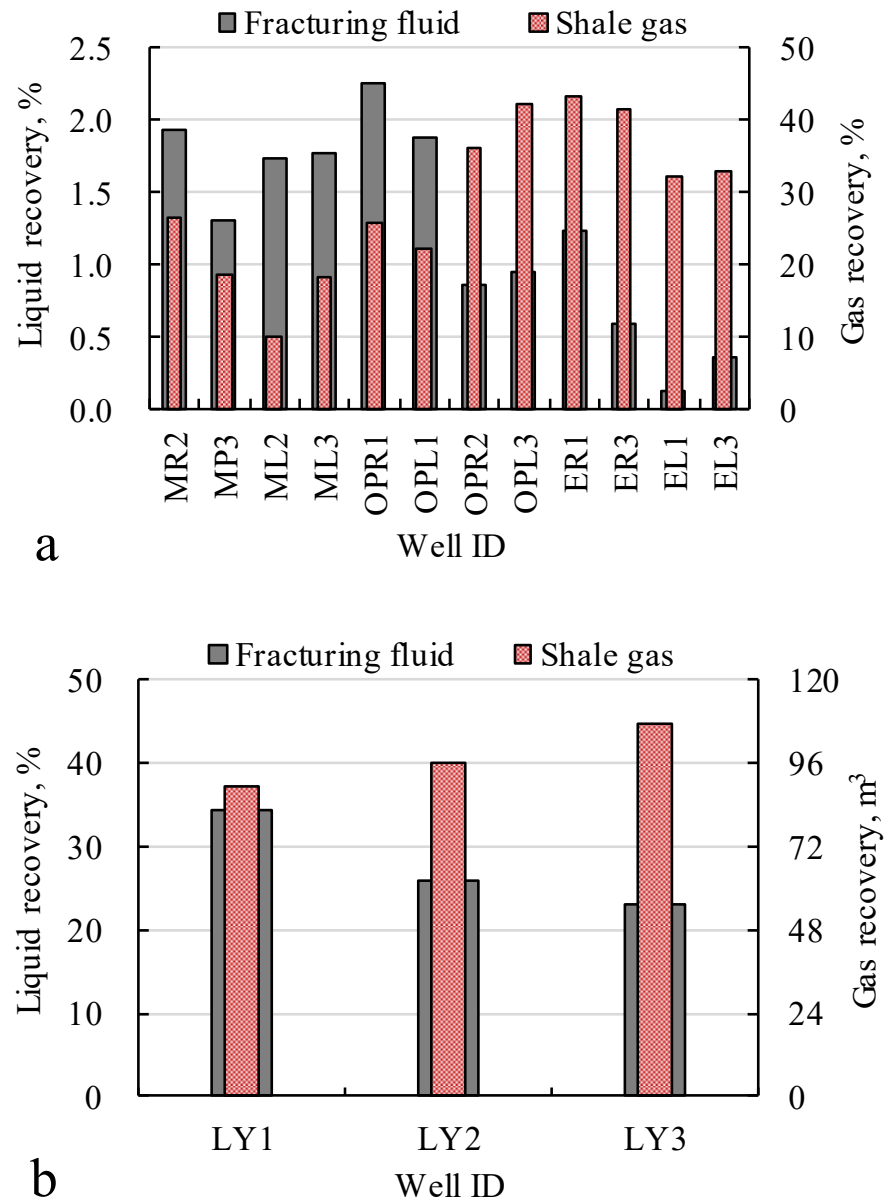
	Stage	Application	Function and Mechanism	Oxidants
Exploration	Kerogen analysis	Selective oxidation	Dissolution	Ruthenium ion (Ru <sup>8+</sup> ), Potassium permanganate (KMnO <sub>4</sub> )
	Clay mineral analysis	Remove OM	Dissolution	H <sub>2</sub> O <sub>2</sub>
	Drilling	Drilling with superheated steam	Generate high temperature	H <sub>2</sub> O <sub>2</sub>
	Completion	Eliminate the filter cake	Dissolution	Highly active oxidant like WF-O etc.
Broken down		Dissolution and Bactericide	Chlorine dioxide (ClO <sub>2</sub> ), H <sub>2</sub> O <sub>2</sub>	
Exploitation	Formation stimulation	Remove formation damage	Generate high temperature	H <sub>2</sub> O <sub>2</sub>
		Fracturing	Generate high pressure	H <sub>2</sub> O <sub>2</sub>
			Dissolution	ClO <sub>2</sub>
			Gel breaking	Ammonium persulfate ((NH <sub>4</sub> ) <sub>2</sub> S <sub>2</sub> O <sub>8</sub> )
			Water flooding	Dissolution
	Enhance oil and gas recovery	Air flooding	Generate high temperature	Oxygen (O <sub>2</sub> )
		Thermal oil recovery	Generate high temperature	H <sub>2</sub> O <sub>2</sub>
		Environmental protection	Wastewater treatment	Dissolution and Bactericide

#### 4. Implementation Clue for Oxidative Fluids Enhancing Organic-Rich Shale Gas Recovery

In recent years, shale gas wells usually exhibit an interesting phenomenon whereby low fracturing fluid recovery is associated with high gas production, while high fracturing fluid recovery forecasts low gas production (Figure 12). The fracture network, as an important sign that determines gas production, may also play a significant role in fluid retention. The more complex a fracture network is for a shale gas well, the higher the gas production will be and the lower the fluid recovery by flowback operations will be (Figure 13). Oppositely, a simple fracture network especially dominated by artificial fractures usually leads to unsatisfactory gas production, while allowing the fracturing fluid retained in the fractures to flow back to ground efficiency due to a relatively low flowback resistance [118].

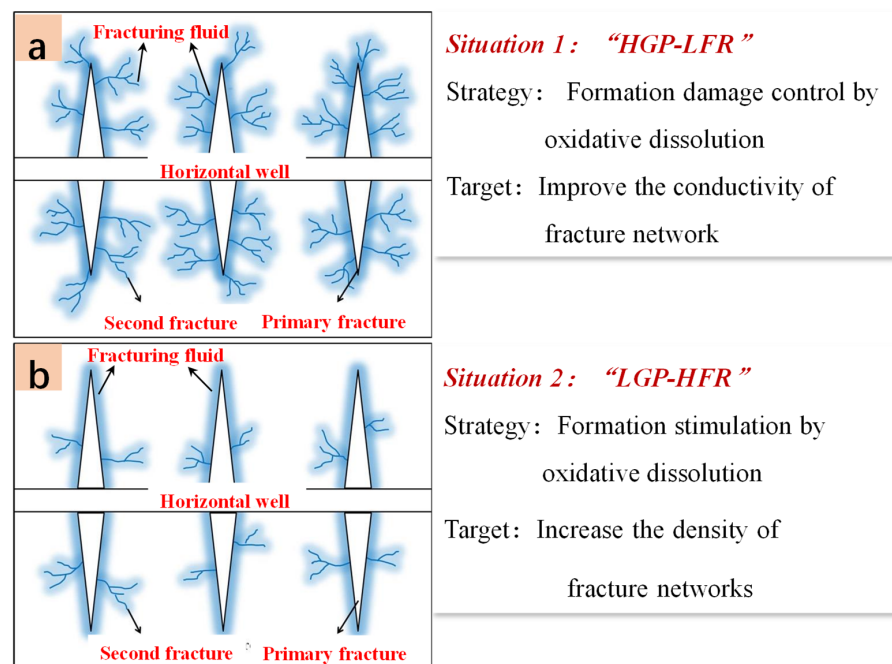
In Figure 13, for shale gas wells with a high gas production and a low fracturing fluid recovery (HGP-LFR), a complex fracture network has been created by hydraulic fracturing. Unfortunately, a large amount of retained fracturing fluid results in some formation damage such as APT in the fracture, impairing the stimulation efficiency by the fracturing operation. Moreover, the stress dependence of unpropped fractures during production has an adverse influence on the conductivity of the fracture network. Therefore, from the perspective of formation damage control, the removal of APT and the reduction of stress dependence of

unpropped fractures are the critical factors for this type of shale gas well to further enhance gas recovery. For shale gas wells with a low gas production and a high fracturing fluid recovery (LGP-HFR), which indicates that the density of fracture networks generated by the fracturing operation is not so high, the increment of the density of fracture networks is the primary goal.



**Figure 12.** Comparison of flowback efficiency and cumulative gas recovery: (a) Shale gas wells from the Horn River basin. Flowback efficiency and cumulative gas recovery were collected after 72 h of opening the wells (after Ghanbari et al. (2013) [21]). (b) Three adjacent shale gas wells in the Sichuan basin experienced the shut-in for 5 days and 15 days, and the production time is 120 days.

Most shale gas wells had a low flowback efficiency for fracturing fluids that ranged from 5–50% of the amount of the injected fluid. Typically, less than 30% of fracturing fluids are recovered during flowback from these wells. Unrecovered fracturing fluid is usually trapped in the pores and microfractures or controlled by other mechanisms [30,31,39,119–121]. Cheng et al. (2020) reported that the imbibition capacity of the shale matrix can be improved up to 11.6–15.3% due to oxidative dissolution [92]. These shale gas wells thereby are further divided into three types to be discussed, as shown in Table 3.



**Figure 13.** Schematic illustration of proposed complex fracture system (a) and simple fracture system (b). The complex fracture system has more secondary fractures which increase the water loss and gas production (after Ghanbari et al. (2013) [21]).

For LGP-HFR, shut-in performance is shown in the increment of the density of fracture networks. Oxidative fluids are added to the pumped fracturing fluid to provide enough oxidants. Shut-in immediately after the fracturing operation contributes to a full reaction between the shale rock and the oxidant. The design of the shut-in time follows a rule that a shale gas well is reopened once fluid pressure in the fracture drops to the minimum horizontal principal stress of the formation, making full use of the effect of loading stress on the generation and propagation of fractures, as described in Section 2.1.

For HGP-LFR, shut-in performance is shown to improve the conductivity of fracture networks. If the flowback efficiency is over 10%, as in HGP-LFR (I), a fast flowback operation in the early stage after the fracturing operation, considering the conventional shut-in strategy, is necessary. This can reduce the water saturation in the fracture and release the APT to some extent. Oxidative fluids are added to the fracturing fluid in the early period of each fracturing stage, in order to ensure that the retained fracturing fluid contains the oxidants after the flowback operations. Meanwhile, in order to avoid the waste of oxidative fluids caused by the flowback operation, the amount of oxidative fluids injected is calculated with the consideration of the flowback efficiency of adjacent wells. This not only can realize the oxidative dissolution of OM in the fracture surface prior to the injection of proppants but also makes full utilization of the effect of retained fluid on maintaining the conductivity of unpropped fractures based on a non-uniform surface-etching profile, as described in Section 2.2.2. If the flowback efficiency is less than 10%, as in HGP-LFR (II), and even no fluid flowback occurs, formation damage control may be particularly important in this case. In addition to the mechanism of oxidizing stimulation mentioned in HGP-LFR (I), redistribution of fracturing fluids retained, which promotes auto removal of APT, is another important mechanism, as described in Section 2.2.1. In this situation, the injection of oxidative fluids and the associated shut-in strategy are the same as those of LGP-HFR. The only difference between them is that no fracturing fluid flowback is present when the shut-in ends for HGP-LFR (II). Furthermore, with the wide distribution of oxidative fluids in the shale matrix, promotion of gas desorption and diffusion by oxidative dissolution, as described in Section 2.2.1, may occur in both LGP-HFR and HGP-LFR.

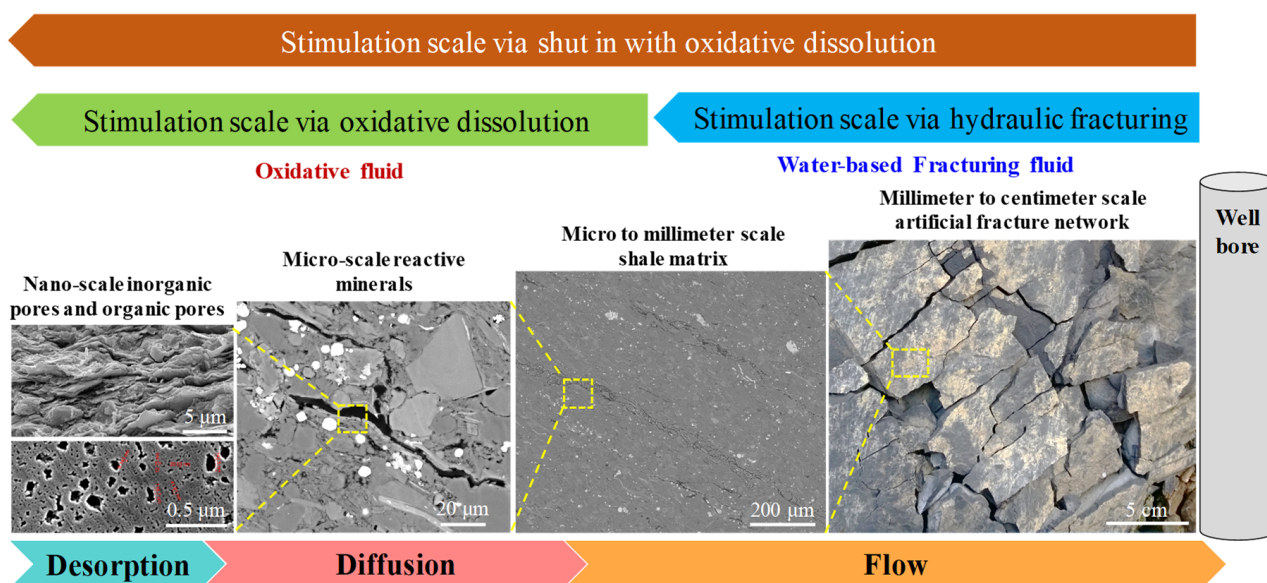
**Table 3.** Shut-in strategy with the application of oxidative fluids in shale gas well.

Type of Shale Gas Wells	Flowback Efficiency of Fracturing Fluid	Engineering Characteristics of Shale Formations	Target of Oxidizing Stimulation	Mechanism of Oxidizing Stimulation	Application of Oxidative Fluids	Shut-in Strategy
LGP-HFR	>30%	(1) Poor hydraulic fracturing performance; (2) Poor water retention capacity.	Formation stimulation: increase the density of fracture networks.	(1) Reduce rock fracture pressure, promoting fracture initiation and propagation; (2) Promote gas desorption and diffusivity.	Injection of oxidative fluids into all the pumped fracturing fluids.	(1) Shut-in immediately after the fracturing operation; (2) Flowback operation once the fluid pressure in the fracture drops to the minimum horizontal principal stress of the formation.
HGP-LFR (I)	<30% and >10%	(1) Good hydraulic fracturing performance; (2) Good water retention capacity.	Formation damage control: improve the conductivity of fracture networks.	(1) Promote proppant migration and distribution; (2) Reduce stress dependence of unpropped fractures;	Injection of oxidative fluids into the partially pumped fracturing fluids.	Fast flowback and then production after the fracturing operation followed the conventional shut-in strategy.
HGP-LFR (II)	<10%	(1) Great hydraulic fracturing performance; (2) Great water retention capacity.		(3) Promote auto removal of aqueous phase trapping; (4) Promote gas desorption and diffusivity.	Injection of oxidative fluids into all the pumped fracturing fluids.	(1) Shut-in immediately after the fracturing operation; (2) Well open and production once the fluid pressure in the fracture drops to the minimum horizontal principal stress of the formation.

## 5. Discussion

A properly shut-in strategy was designed for shale gas wells for the enhancement of gas production. Currently, shut-in performance usually depends on the combined effects of formation stimulation and damage control behind the fracturing operations. This is attributed to the increment of the density of fracture networks, the improvement of fracture network conductivity, and the promotion of gas desorption and diffusivity. In another words, shut-in has a positive effect on further improving the shale gas transport path from the matrix pore to the wellbore after hydraulic fracturing operations.

Oxidizing stimulation for organic-rich shale was originally proposed for further enhancing the methane production from the matrix in post-fractured shale gas wells, as shown in Figure 14 [14]. Dissolution of shale rock components such as OM, pyrite and associated, induced pores and microfractures with oxidative fluids, contributing to a higher desorption or diffusion of adsorbed and free gas [13,63]. Furthermore, this study attempted to demonstrate the mechanisms of oxidative dissolution increasing shut-in performance, to evaluate the feasibility of using oxidative fluids, and finally, to develop an implementation clue for enhancing organic-rich shale gas recovery. This indicates that the increment of shut-in performance through oxidative dissolution is a new insight into enhancing organic-rich shale gas recovery.



**Figure 14.** Schematic illustration of oxidative dissolution coordinated hydraulic fracturing stimulation in organic-rich shale reservoirs.

However, oxidizing stimulation is a new field much of whose theoretical knowledge needs to be explored and established. On the one hand, although the mechanisms of oxidative dissolution increasing shut-in performance are clear, associated laboratory experiments and the simulation study at field-scale to verify its effectiveness are still lacking. For example, taking into consideration the change in the chemical field, the temperature field, the flow field, and the pressure field during the shale and oxidative fluids reaction, the effect of multi-field coupling on oxidation-increase permeability of shale rocks still needs to be evaluated. On the other hand, the injection of oxidative fluids into the formation possibly induces some formation damages which have an adverse influence on the enhancement of shale gas recovery. For example, the shale and oxidative fluids reaction produces some precipitations such as  $\text{Fe}_2\text{O}_3$ ,  $\text{Fe}(\text{OH})_3$ ,  $\text{FeCO}_3$  (siderite),  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  (dihydrate gypsum), etc., as well as solid particles of shale debris, which will block the pore or fracture, resulting in a decrease of rock permeability [62,63,67,122,123]. In response, You et al. (2021a; 2021b) proposed the evaluation of oxidation sensitivity for the oxidizing stimulation in organic-rich shale, which can play a significant role in preventing damage and converting the harm



into a benefit [67,123]. Moreover, oxidative dissolution-induced potential groundwater pollution is another problem that should be taken into further consideration [62]. Therefore, in order to establish and perfect the oxidating stimulation technology, there is still much work to do in terms of the evaluation of the engineering adaptability of oxidative fluids, enhancing organic-rich shale gas recovery, and the development of an oxidative fluid satisfying the standard of engineering applications.

## 6. Conclusions

Oxidative fluids, which contribute to the increment of the density of fracture networks, improve fracture network conductivity, promote gas desorption and diffusion, can play an important role in the enhancement of shut-in performance compared with the conventional fracturing fluid.

(1) Oxidative dissolution shows a potential to fully leverage the retained fracturing fluid through the designed shut-in strategy, which aims to convert the detrimental fracturing fluid retention into a beneficial force to enhance the organic-rich shale gas recovery with a lower cost.

(2) The application of oxidative fluids in shale gas wells to enhance organic-rich shale gas recovery is a new domain. Much theoretical knowledge needs to be explored and established. The evaluation of engineering adaptability of oxidative fluids for enhancing organic-rich shale gas recovery and the development of oxidative fluids satisfying the benchmark of engineering applications is necessary for the next stage.

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