Contents lists available at ScienceDirect

Petroleum

South Hard Petroleum

journal homepage: http://www.keaipublishing.com/en/journals/petroleum

Effects of inhibitor KCl on shale expansibility and mechanical properties

Xiangchao Shi^{a,c,*}, Lei Wang^a, Jianhua Guo^b, Qiang Su^b, Xiao Zhuo^a

^a State Key Laboratory of Oil and Gas Reservoir Geology and Exploitation, Southwest Petroleum University, Chengdu, 610500, China

^b Engineering Technology Research Institute of South West Oil and Gas Field Company, CNPC, Chengdu, 610000, China

^c State Key Laboratory of Geomechanics and Geotechnical Engineering, Institute of Rock and Soil Mechanics, Chinese Academy of Sciences, Wuhan, 430071, China

ARTICLE INFO

Keywords:

Inhibitor

Expansibility

Bedding fissures

Mechanical properties

KC1

ABSTRACT

The expansibility and mechanical properties of shale are significantly influenced by water-based muds (WBMs); thus, it is necessary to mitigate this effect to avoid borehole instabilities in drilling operation. Potassium chloride (KCl) is usually used as inhibitor to reduce hydration of shales. In this study, we investigated the inhibitory efficiency of KCl on shale through a series of experiments, including dynamic linear swelling (DLS) tests and uniaxial compressive strength (UCS) tests, to provide reference for the design of WBMs. These tests were conducted on shale samples soaked in KCl solution for 24, 48, 72, and 96 h with saline concentrations of 0%, 2%, 4%, 6%, and 8%. Experimental results show that samples with microcracks and bedding fissures have the highest swelling increase and the largest strength reduction after immersion in solution. The swelling potential decreased with increasing KCl concentration. In addition, KCl exhibited a certain inhibitory effect on the weakening of the mechanical properties of samples. An increase in the KCl concentration increases the compressive strength and elastic modulus, and decreases the Poisson's ratio. However, in terms of homogeneous samples, the UCS test results show that exposure to water is weakly related to weakening of the mechanical properties of shale samples. We found that immersing the shale in KCl solution for a longer time decreases the compressive strength, increases the Poisson's ratio, and decreases the elastic modulus.

1. Introduction

As one of the most important unconventional oil and gas resources, shale gas has a significant potential for wide distribution and mining. In 2011, the US Energy Information Administration (EIA) evaluated global shale gas resources. The result shows that China has the world's largest recoverable shale gas, which is mainly located in the Sichuan Basin, Erdos Basin, and Tarim Basin [1]. Although China's shale gas reserves are abundant, the drilling process of shale reservoir faces a series of engineering problems, especially shale formation collapse, which have seriously plagued shale gas exploitation and even the petroleum industry [2].

To solve wellbore instability problems in the shale reservoir drilling process, potassium chloride (KCl) has been incorporated into the drilling mud as drilling fluid additive [3-8]. Scholars have carried out many studies on the effects of potassium ions on the expansibility and mechanical properties of shale in solution [9-13]. Most studies found that K+ cations' ability to lower hydration tendency of smectite can significantly reduce the swelling tendency of shale. Owing to their small size, the K+ ions can induce inhibition as they fit into the

montmorillonite structure [14,15]. Another inhibition mechanism that uses heavy brines to increase the water phase ionic concentration was proposed by Gomez et al. [16]; thus, high ionic concentration reduces hydration by minimizing the osmotic effect. Yang et al. [17] conducted an experiment to investigate changes in the mechanical properties of Longmaxi Formation shale after saturation in different saline solutions with different concentrations. The results show that potassium chloride solution with a mass fraction of 20% can effectively reduce pore pressure transfer capability and prevent water from infiltrating the rock. Similarly, Zhang et al. [18] reported that high concentrations of electrolytes can reduce swelling by destroying the hydration shell that surrounds smectite particles. Yan et al. [19] found that KCl zwitterionic polysulfide drilling fluid can inhibit the expansion and reduction of the compressive strength of rock after performing drilling fluid tests in sand and mudstone. AI-Bazali [20] investigated the effects of water and ion intrusion on the compressive strength of shale through biaxial stress testing. The results show that the adsorption of ions changes the shale's fabric structure and increases the shale strength. Wang et al. [21] concluded that fluid immersion leads to the extension of cracks in shale, which in turn serves to reduce the strength of shale. In addition, results

E-mail address: sxcdream@163.com (X. Shi).

https://doi.org/10.1016/j.petlm.2018.12.005



Peer review under responsibility of Southwest Petroleum University.

^{*} Corresponding author.

Received 22 September 2018; Received in revised form 30 November 2018; Accepted 11 December 2018

^{2405-6561/} Copyright © 2019 Southwest Petroleum University. Production and hosting by Elsevier B. V. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/BY-NC-ND/4.0/).

of shale immersion experiments by Liang et al. [22] show that KCl solution has a certain inhibitory effect on shale hydration.

Based on previous studies, it is clear that there is a relationship between shale swelling, mechanical properties, and KCl concentration. In other words, KCl can inhibit shale hydration and effectively solve the problem of wellbore instability during drilling. Therefore, the effect of KCl concentration on the swelling and mechanical characteristics of shale after water absorption should be clarified. In this study, the swelling and mechanical properties of Longmaxi shale in KCl solutions of different concentrations for different test durations were investigated using uniaxial compressive strength (UCS) tests together with dynamic linear swelling (DLS) tests. The findings will provide reference for research on shale wellbore stability, which will enhance shale drilling efficiency.

2. Experimental materials and methods

2.1. Materials

2.1.1. Samples preparation

We tested shale samples collected from two different areas in the southwestern edge of Sichuan Basin, namely Shizhu and Pengshui in Chongqing Municipality. The blocks were cored perpendicular to the bedding and cut into 25-mm diameter cylinders. The length of each sample was selected as 50 mm to ensure a height-to-diameter ratio of 2:1. Two round surfaces were carefully ground to make smooth faces. Then, rock samples with no significant difference in physical properties were selected and heated at 105 °C in an electrothermal constant temperature drying box until the weight of the sample remained constant. Before the experiments, all shale samples were double sealed and stored in a dry room. The mineralogical composition of shale obtained from one sample cored in the same block was tested using a Dutch X'Pert Pro X-ray diffractometer. The results are listed in Table 1, indicating that the Longmaxi Formation shale is predominately composed of quartz, clay minerals, calcite, and dolomite. In general, the samples' black siliceous (quartz) and calcareous (calcite) contents are high, whereas the clay mineral content is low. In particular, the clay mineral contents of Shizhu and Pengshui shale are 10.65% and 30.12%, respectively, and the predominant clay mineral is illite.

2.1.2. Experimental devices

The swelling tests were primarily carried out using ZNP-01 shale dilatometers with an accuracy and range of 0.01 mm and 10 mm, respectively to directly record the expansion of the rock samples. The triaxial stress–strain curve for rock was mainly derived based on the results of the rock triaxial mechanical test system of the State Key Laboratory of Oil and Gas Reservoir Geology and Exploitation of China.

2.2. Experimental methods

Twenty samples from Shizhu were divided into five groups, similar to the samples from Pengshui. One sample without fluid saturation was used for the control test (see Table 2).

First, the dial gauge was adjusted to zero after placing the samples in measuring cylinders. Before the soaking process, the states of the samples were recorded using a camera. The measuring cylinders were

Table 2

Arrangement of sample saturation.

Soaking condition	Number of samples		
KCl Concentration (% weight)	Time (h)		
Dry sample	-	1	
0	24/48/72/96	4	
2	24/48/72/96	4	
4	24/48/72/96	4	
6	24/48/72/96	4	
8	24/48/72/96	4	



Fig. 1. Experimental flow chart.

then placed in containers with solution of KCl. The concentrations of KCl solution were 2%, 4%, 6%, and 8% by weight, and the immersion periods were 24, 48, 72, and 96 h. The amounts of swelling of the samples were recorded every 1 h within 24 h, and every 6 h for more than 24 h. When the experimental periods have been satisfied, the samples were removed and sealed with polyethylene bags for UCS tests. The UCS tests were conducted using a rock triaxial mechanical test system. The loading rate was 0.1 mm/min for all the tested shale samples. The experimental process is shown in Fig. 1.

3. Results and discussions

The results obtained from the experiments provide some important conclusions on the effect of different concentration of solutions for different test durations on the amount of swelling and mechanical properties of shale samples. The results are discussed under two headings: (1) swelling variation and (2) mechanical properties.

3.1. Swelling variation

The axial expansion of each sample was tested at normal atmospheric temperature and pressure while absorbing fluids. When the difference in the last two recordings was within 0.01 mm, the next test can be conducted. The amount of swelling of the shale sample was obtained by reading the dial indicator.

Fig. 2 shows the variation of shale swelling in different

Table 1

Mineral composition of Longmaxi Formation shale in Sichuan Basin (unit: %).

1	v									
Sampling location	ation Clay rock			Brittle rock	Brittle rock			Other ore		
	I	I/S	С	Quart	Orthodox	Plagioclase	Calcite	Dolomite	Pyrite	
Shizhu County Pengshui County	8.47 14.63	1.14 7.44	1.04 8.05	52.59 36.96	0.59 2.07	1.48 8.49	26.72 4.86	6.37 15.47	1.6 2.03	

I-illite; I/S-illite/smectite; C-chlorite.



Fig. 2. Swelling of shale samples with water and KCl imbibition.



Fig. 3. State of representative samples after immersion.

concentrations of KCl solutions. The samples collectively show a small amount of swelling for a low clay content. Water immersion caused the largest expansion in all the fluid-absorbed samples. Moreover, increasing KCl concentration in the solution leads to a reduction in the amount of swelling, indicating that KCl plays a role in inhibiting shale expansion. In the first 48 h, the expansion of the Pengshui samples stabilized, whereas the Shizhu shale samples continued to expand throughout the test period. Typical macroscopic changes of shale samples after the swelling tests are shown in Fig. 3. It can be observed that the development of bedding fissures within the Shizhu shale is more significant than that within the Pengshui shale. This phenomenon is related to the larger final expansion of the Shizhu shale, which has a lower clay content. The result indicates that the effects of micro-cracks on the expansion of shale are significant.

3.2. Mechanical properties

3.2.1. Stress-strain curve analysis

Fig. 4 shows the stress-strain curves of the Longmaxi Formation shale samples in different concentrations (0, 2%, 6%, and 8%) of KCl solutions for 96 h. The result shows that for all shale samples, the axial strain is higher than the lateral strain for the same test conditions. Moreover, water-absorbed samples have higher axial and lateral strains than other samples, whereas the axial and lateral strains for dry shale samples are the lowest, except for the Shizhu sample in KCl concentration of 2% by weight and the Pengshui sample in KCl concentration of 8% by weight. It is clear that a higher KCl concentration results in a lower strain variation. This is in accordance with swelling results showing that samples absorbed in higher KCl concentrations have lower amount of swelling.

According to Fig. 4, hydration expansion occurred in Shizhu

samples in KCl solution, and the stress–strain curves of the solutionabsorbed samples show a long compaction stage and a slow transition into the elastic deformation stage. Various degrees of bedding were observed after the swelling tests due to the anisotropy of the Shizhu shale, which causes irregularity at the beginning of the stress–strain curves. However, unlike the Shizhu samples, the curves of the Pengshui samples show a shorter compaction stage and a rapid transition into the elastic stage. Because the samples were not affected by the anisotropy of the Pengshui shale, the stress–strain curves are more regular.

3.2.2. Feature parameter

3.2.2.1. Uniaxial compressive strength. The intact samples' UCS of Shizhu and Pengshui are 172.44 MPa and 111.29 MPa, respectively. Fig. 5 shows results of UCS for the solution-soaked condition. It is clear that the UCS values of the Shizhu and Pengshui samples are mostly distributed between 100 MPa and 140 MPa, and 80 MPa and 120 MPa, respectively. We can conclude that the average UCS values of Shizhu and Pengshui samples are approximately 35% and 9% lower than that of the intact samples, respectively. The formation of bedding and fracture has a greater impact on the shale strength than hydration of clay minerals.

In terms of the solution-soaked samples, the UCS of the shale slightly increased with increasing KCl concentration, except for the heterogeneous sample. During the imbibition experiments, the flow of water and ions into the shale produces more micro cracks, which causes bedding fissures and significantly decreases the average UCS value of the Shizhu samples. This is mainly due to different degrees of bedding fissures of the Shizhu samples, which results in dispersed distribution of their UCS values in the samples. By contrast, the overall UCS values of Pengshui samples were not significantly reduced after water immersion. Bedding fractures were only observed in the Pengshui samples soaked



Fig. 4. Stress-strain curves for samples in KCl solutions.



Fig. 5. Effects of KCl concentration on shale samples' UCS values.



Fig. 6. Effects of KCl concentration on shale samples' Poisson's ratio.



Fig. 7. Effects of KCl concentration on the elastic modulus of shale samples.

in 8 wt% and 2 wt% KCl solution for 96 h, which is presumed to have contributed to data irregularity. In addition, the UCS of shale slightly increases as the KCl concentration of the immersion solution increases. In summary, increasing bedding fissures and micro fractures strengthened hydration effect. Moreover, even with the inhibitory effects of KCl, the UCS of shale tends to decrease with increasing micro-cracks and bedding fissures.

3.2.2.2. Poisson's ratio. The Poisson's ratio is an elastic constant defined as the ratio of the lateral contraction to elongation in an infinitesimal uniaxial extension of a homogeneous isotropic body. In other words, it reflects the transverse deformation of materials. Fig. 6 shows the Poisson's ratios of Shizhu and Pengshui samples soaked in different concentrations of KCl solutions with different test durations.

In general, the Poisson's ratio of shale samples decreases with increase in KCl concentration. The Poisson's ratios of the Shizhu samples treated with KCl were in the range of 0.18–0.28, whereas those of the Pengshui shale samples were in the range of 0.25–0.35. The Pengshui shale exhibited higher average Poisson's ratio after soaking owing to its higher expansible clay content. It is clear that the Shizhu shale soaked in aqueous solution have higher Poisson's ratio of the soaked Shizhu samples is not uniform owing to its anisotropy. The Poisson's ratios of the Pengshui samples have obvious regularity, decreasing with increasing KCl concentration. Furthermore, the Poisson's ratio slightly increased as the soaking time increased. These results demonstrate that the effects of KCl dissolved in an aqueous solution on the Poisson's ratio of shale are significantly influenced by bedding fissures and clay mineral contents.

3.2.2.3. Elastic modulus. Elastic modulus is another important characteristic of shale mechanics, which represents the stiffness of rock. Fig. 7 shows the elastic modulus of shale samples in KCl solutions.

The influence of KCl dissolved in an aqueous solution on the elastic modulus of shale is the same as that on the Poisson's ratio, as shown in Fig. 7. The elastic moduli of the soaked Shizhu samples were mostly within the range of 13-17.5 GPa, whereas those of the soaked Pengshui samples were mostly within the range of 12-15 GPa. Thus, the average elastic modulus of the Shizhu shale is slightly higher than that of the Pengshui shale. However, it was observed that the elastic modulus of the Pengshui shale is less influenced by the KCl concentration compared to that of the Shizhu shale. The average elastic modulus of the Shizhu solution-soaked samples decreased by 33.3% compared to that of the dry sample. Furthermore, the elastic modulus of soaked samples increases with increasing KCl concentration because the development of bedding fissures in the Shizhu shale may have strengthened the inhibitory effects of KCl. On the other hand, the average elastic modulus of the Pengshui solution-soaked samples decreased by 16.7% compared to that of the dry sample. Although the elastic modulus exhibited minimal dependence on the KCl concentration, it generally decreased with increase in the soaking time.

4. Conclusions

- (1) The clay mineral content of the Longmaxi Formation Shizhu and Pengshui shales is relatively low, and mostly includes illite, a small amount of expansive clay mineral such as montmorillonite and a small amount of illite-smectite mixed-layer minerals. This composition ensures that the shales of the Longmaxi Formation exhibits strong brittle characteristics.
- (2) Although the amount of swelling of the Longmaxi Formation shale slightly decreased with increase in the KCl concentration in solution, the relative expansions of samples were small due to low clay content. In addition, the amount of swelling is also significantly affected by bedding fissures.
- (3) Under uniaxial compression, the compaction stage of the

stress-strain curve of the Longmaxi Formation shale samples is short, and the curve shows a rapid transition into the elastic deformation stage. In addition, rock has high UCS and strong elastic deformation characteristics such that it quickly loses its pressurebearing capability after failure, which makes it susceptible to elastic-brittle failure.

- (4) The UCS of shale increases slightly with increasing KCl concentration. However, when shale is soaked, more cracks will appear, which further weakens the compressive strength of the rock. Similarly, although the Poisson's ratio of shale decreases with increasing KCl concentration, it is significantly affected by the development of rock bedding. Moreover, for the Shizhu shale with significantly developed bedding fissures, the elastic modulus of the rock showed a slight tendency to increase with increase in the KCl concentration, whereas the more homogeneous Pengshui shale was not affected by the concentration of KCl.
- (5) Overall, the clay content of shale in the research area is low; thus, the effect of hydration is limited, and the effect of KCl is not obvious. The clay content of the Pengshui shale was higher, but the inhibitory effect of KCl on hydration was not significant. By contrast, more micro-cracks and bedding fissures occurred in the Shizhu shale after soaking in solution, which obviously weakens the mechanical characteristics of shale. Therefore, we believe that the drilling fluid used in this type of shale formation should improve the plugging performance of the drilling fluid and prevent secondary damage caused by invading fluid, which is key to the design of the drilling fluid.

Acknowledgements

This work was financially supported by the Foundation of National Natural Science Foundation of China (No. 511774248 and 51504208), the Open Research Fund of the Key Laboratory of Deep Undergroud Science and Engineering(Sichuan University), Minstry of Education, China and the Open Research Fund of the State Key Laboratory of Geomechanics and Geotechnical Engineering, Institute of Rock and Soil Mechanics, Chinese Academy of Sciences, China (No. Z016013).

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.petlm.2018.12.005.

References

- U.S. Energy Information Administration, Annual energy outlook 2011 with projections to 2035, (2011) http://www.eia.gov/forecast/aeo/df/0383(2011a).pdf/, Accessed date: 16 August 2011.
- [2] S. Cui, F. Ban, G. Yuan, Status quo and challenges of global shale gas drilling and completion, Nat. Gas. Ind. 31 (2011) 72–75 https://doi.org/10.3787/j.issn.1000-0976.2011.04.017.
- [3] Y. Liu, X.F. Li, P.C. Qing, Use of sea water potassium chloride drilling fluid in evaluation well xianhe-4, Oilfield Chem. 22 (2005) 101–103 https://doi.org/10. 3969/j.issn.1000-4092.2005.02.002.
- [4] R.K. Clark, R.F. Scheuerman, H. Rath, H.G. Van Laar, Polyacrylamide/Potassium-Chloride Mud for Drilling Water-Sensitive Shales, J. Petrol. Technol. 28 (1976) 719–727 https://doi.org/10.2118/5514-PA.
- [5] Q.W. Huang, J.W. Xu, X.H. Wang, G.W. Hu, C.F. Chen, Application of Inhibiting Clay Hydration and Expansion by Organic Salt, Oilfield Chem. 30 (2013) 496–499 https://doi.org/10.19346/j.cnki.1000-4092.2013.04.005.
- [6] L. Zhao, Y. Guo, J. Li, J. Yu, W. Yang, Application of organic KCl high-density drilling fluid technology in well Jinhe-1, Drill. Fluid Complet. Fluid 28 (2011) 78–80 https://doi.org/10.3969/j.issn.1001-5620.2011.01.024.
- [7] Y. Qu, X. Lai, L. Zou, Y. Su, Polyoxyalkyleneamine as shale inhibitor in water-based drilling fluids, Appl. Clay Sci. 44 (2009) 265–268 https://doi.org/10.1016/j.clay. 2009.03.003.
- [8] R. Gholami, H. Elochukwu, N. Fakhari, M. Sarmadivaleh, A review on borehole instability in active shale formations: Interactions, mechanisms and inhibitors, Earth Sci. Rev. 177 (2018) 2–13 https://doi.org/10.1016/j.earscirev.2017.11.002.
- T. Al-Bazali, The impact of water content and ionic diffusion on the uniaxial compressive strength of shale, Egypt, J. Petrol. 22 (2013) 249–260 https://doi.org/ 10.1016/j.ejpe.2013.06.004.

- [10] B. Bostrom, G. Svano, P. Horsrud, A. Askevold, The Shrinkage Rate of KCL-Exposed Smectitic North Sea Shale Simulated by a Diffusion Model.SPE-47254-MS, (1998) https://doi.org/10.2118/47254-MS.
- [11] P. Horsrud, B. Bostrom, E.F. Sonstebo, R.M. Holt, Interaction between shale and water-based drilling fluids: laboratory exposure tests give new insight into mechanisms and field consequences of KCl contents, (1998) SPE-48986-MS https:// doi.org/10.2118/48986-MS.
- [12] Huadi, F., Aldea, C., MacKereth, B. M., Mukhlis, T. Successful KCl Free Highly inhibitve and Cost Effective WBM Applications, Offshore East Kalimantan, Indonesia. SPE-132690-MS. https://doi.org/10.2118/132690-MS.
- [13] H. Zhong, W. Huang, Z. Qiu, M. Hang D, F. Wang, Experimental Study of the Shale Inhibition Between Polyamine and Potassium Chloride vol. 34, (2012), pp. 150–156 https://doi.org/10.3863/j.issn.1674-5086.2012.03.023.
- [14] A.T. Bourgone Jr., K.K. Millheim, M.E. Chenevert, F.S. Young Jr., Applied Drilling Engineering, Society of Petroleum Engineers, Richardous, Texas, USA, 1986.
- [15] M.K. Al-Arfaj, M. Amanullah, A.S. Sultan, M.E. Hossain, A. Abdulraheem, Chemical and Mechanical Aspects of Wellbore Stability in Shale Formations: A Literature Review, Society of Petroleum Engineers, 2014 SPE-171682-MS https://doi.org/10. 2118/171682-MS.
- [16] S.L. Gomez, A. Patel, Shale Inhibition: What Works? Society of Petroleum Engineers, SPE-164108-MS, (2013) https://doi.org/10.2118/164108-MS.

- [17] X. Yang, Z. Shang, Y. Shi, Y. Peng, Y. Yue, S. Chen, G. Jiang, J. Cai, Influence of salt solutions on the permeability, membrane efficiency and wettability of the Lower Silurian Longmaxi shale in Xiushan, Southwest China, Appl. Clay Sci. 158 (2018) 83–93 https://doi.org/10.1016/j.clay.2018.02.006.
- [18] F. Zhang, P.F. Low, C.B. Roth, Effects of Monovalent, Exchangeable Cations and Electrolytes on the Relation between Swelling Pressure and Interlayer Distance in Montmorillonite, J. Colloid Interface Sci. 173 (1995) 34–41 https://doi.org/10. 1006/jcis.1995.1293.
- [19] J. Yan, J. Luo, A comprehensive method for evaluating the anti-sloughing effectiveness of drilling fluids, J. Univ. Petrol. China 23 (1999) 31–34 https://doi.org/ 10.3321/j.issn:1000-5870.1999.01.009.
- [20] T.M. AL-Bazali, The consequences of using concentrated salt solutions for mitigating wellbore instability in shales, J. Petrol. Sci. Eng. 80 (2011) 94–101 https:// doi.org/10.1016/j.petrol.2011.10.005.
- [21] Y. Wang, J. Xu, C.G. Mei, B. Ou, L. Li, Chemical and Mechanical Properties of Brittle Fractured Mud Shale, J. Oil Gas Technol. 33 (2011) 104–108 http://doi.org/10. 3969/j.issn.1000-9752.2011.06.022.
- [22] L. Liang, J. Xiong, X. Liu, Effects of hydration swelling and wettability on propagation mechanism of shale formation crack, Petrol. Geol. Exp. 36 (2014) 780–786 http://doi.org/10.11781/sysydz201406780.