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Experiments to Evaluate Swelling Inhibition of Coal with High Salinity by Sodium Dodecyl Benzene Sulfonate Concentration

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Abstract: *We studied coal specimens from the #3 coalbed of Shaoyang, Hunan province, to try to understand the processes the inhibited expansion of the coal beds. Experiments were designed to test the effect of the concentration of sodium dodecycl benzene sulfonate (SDBS) solutions on the swelling inhibition of the coalbed with five different high water salinities containing $CaCl_2$, and also the synergistic behavior between the SDBS, the Ca^{2+} and clay mineral were analyzed. The results indicated that: the swelling of the coals was mainly affected by its water salinity when either fresh water or solutions with low SDBS concentration was used as fracturing fluid additives. The coals with higher water salinity had the weaker hydration and the better relative stability. Besides, due to the attraction between Ca^{2+} and clay unit layer as well as the complexation reaction between Ca^{2+} and SDBS, the optimal SDBS solution concentration for the coals with five different water salinities（1000 mg/L to 5000 mg/L）are 200mg/L, 400mg/L, 200 mg/L, 300 mg/L, and 100mg/L, respectively, and their swell increment was 0.18 mm, 0.1 mm, 0.11 mm, 0.13 mm, 0.1mm respectively after 8 h. Furthermore, Within the scope of the water salinity of coalbed in this experiment, the expansion ratio of the samples can be determined by the fitting formula under the SDBS solution concentrations, thereby quantitatively predict the damage to coals caused by the fracturing fluid and then adjust the fracturing fluid formula to reduce the damage to the coalbed methane reservoir.*

Keywords: *swelling inhibition; coal rock; salinity; sodium dodecycl benzene sulfonate; expansion ratio*

1. Introduction

Reservoir permeability is an important factor in coalbed methane production [1-3] during the exploitation process. Most of the coalbed methane reservoirs need to be fractured to improve production due to the generally ultra-low permeability of coalbeds [4-5] in China. It is found that the water salinity of the Duanpiqiao coalbed in Shaoyang, Hunan is very high[6], generally in the 1000-3000 mg/L and some values reach 4000 mg/L, with the highest 4782mg/L. In addition, the type of coalbed water is mainly the calcium chloride-type according to the Surin classification. Therefore, the filtrate can easily seep into the coal and react with the reservoir mineral ions [7-8] in the hydraulic fracturing process.

For the incompatibility between fracturing fluid and formation water, most researches [9-10] use the formation water to prepare fracturing fluid in order to balance the activity between fracturing fluid and formation water. This can prevent the filtrate from seeping into the strata. However, compared with fresh water, formation water has high salinity, complex ionic composition, high organic matter content, and large numbers of saprophytic bacteria [11-12], which can cause problems in the preparation of fracturing fluid.

Surfactant plays an important role in the fracturing process and anionic surfactant is widely used [13] because of its higher surface activity, better temperature resistance and low cost, especially the sodium dodecyl benzene sulfonate (SDBS). To adjust to formations with different water salinities, studies have been primarily concerned about the salt resistance of surfactants [14-15] and most previous studies have focused more on the influence of inorganic salt on the macroscopic properties of surfactant solution. Less research has been done on the micro-interaction between surfactants and inorganic salt. And the synergistic effect of the surfactant and inorganic salt on the inhibition of coal expansion have not been reported, but previous studies have showed that the influence of surfactant on rock was often affected by the inorganic ion from the formation water [16].

Therefore, a coalbed at Duanpiqiao in Shaoyang, in Hunan province was taken as a research target, and laboratory test is designed to test the influence of the concentration of anionic surfactant solutions on the swelling inhibition of the coalbed with five different

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water salinities on the chemical micro-level in this paper. It is useful to regulate and control the fluid fracturing system that is suitable for reservoirs with different salinities and also provide a research foundation for the exploitation of coalbed methane.

2. Experiments

2.1 Experimental devices and materials

The schematic diagram of "CPZ-Ⅱ"shale dilatometer with dual channel is shown in Fig. 1. This apparatus is composed of main engine (including sample cups, beaker, sensor, digital display) and the sampled data system.

The other apparatus was X-ray diffractometer, electric-blast drying oven, balance and "ZNGJ-2" digital-display homogenizer.

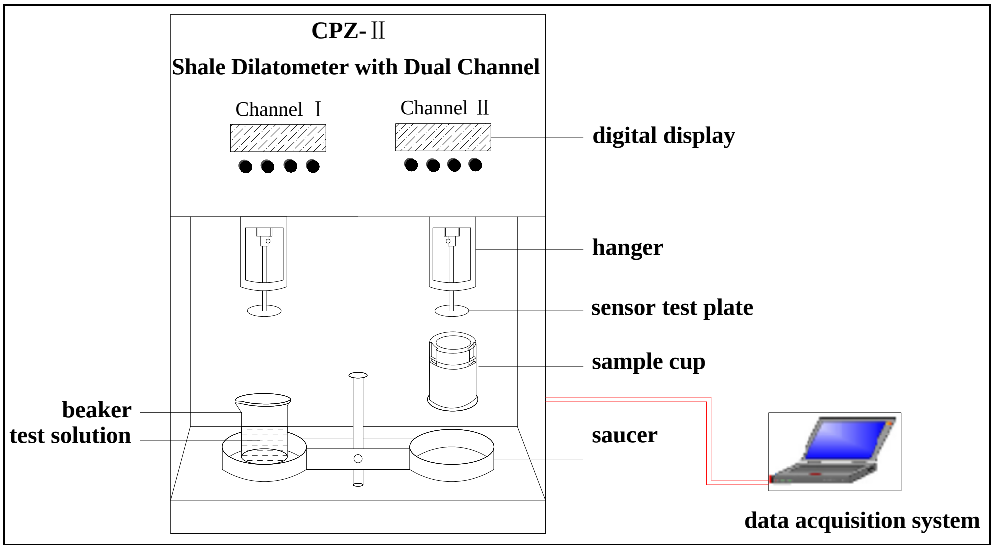


Fig. 1 *Schematic diagram of "CPZ-Ⅱ" shale dilatometer with dual channel*

The test materials were sodium dodecyl benzene sulfonate (SDBS) (Fig. 2), anhydrous $CaCl_2$ (analytically pure), distilled water, coal specimens.

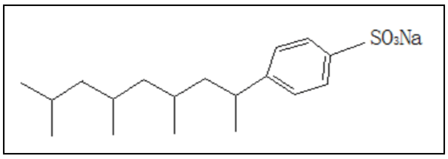


Fig. 2 *Structural formula of sodium dodecyl benzene sulfonate (SDBS)*

2.2 Coal samples

The studied coal specimens were taken from coalbed of Shaoyang, Hunan province (Fig. 3); and the raw coal is shown in Fig. 4(a). The coals were smashed, sieved, dried in an electric-blast drying oven, and then stored in the dryer before the test. The 100 ~ 200 mesh coal samples are shown in Fig. 4(b).

The mineral analysis of the coal is shown in Table 1. As we can see that the inorganic components of coals consists of rich montmorillonite, and small amounts of quartz, pyrite, and anatase. This meant that reservoirs were highly sensitive to external fluid like fresh water.

2.3. Methods

(1) Specimen made process: In order to make the pulverized coals with different salinities, use distilled water to prepare five different kinds of water with different salinities (water type belongs to $CaCl_2$: 1000 mg/L, 2000 mg/L, 3000 mg/L, 4000 mg/L, 5000 mg/L). And then add the experimental water into the dry pulverized coals to make its moisture content become 10 %. Finally, keep the processed pulverized coals in sealed bags.

(2) Swelling experiments: At first, put the well-compacted coal samples (as shown in Fig. 4 (c)) on the hanger, together with the sample cups. At this time, the coal samples will be exposed to sensor test plate. Then adjust zero and put the test solution into beaker, lift the beaker to make test solution slowly seep into coal cups and be exposed to coal samples. Next, remove the sample cups after 8 hours and collect data from the data acquisition system.

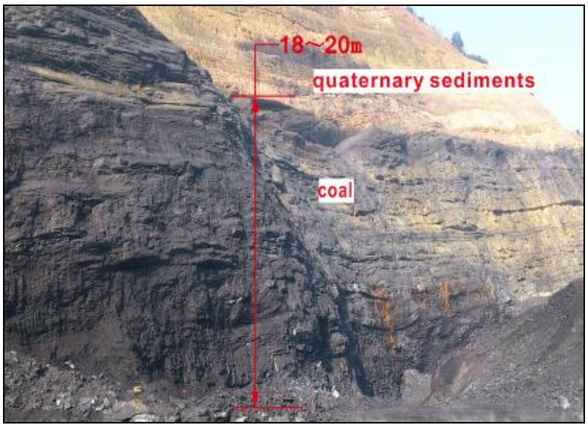


Fig. 3 *General situation of stratum*

(a) (b) (c)

Fig. 4 *Coal specimen*
((a),(b) and (c)were the raw coal, 100 ~ 200 mesh coal samples and well-compacted coal specimen, respectively)

Table 1 *X-ray diffraction semi-quantitative analysis results of Coal rock mineral composition*

Rock specimen	A	V	O	H	M	W	L	N
Coal	20%	13%	16%	12%	25%	11%	3%	The rest

Note: The mineral of samples are marked as: A—pyrophyllite; V—quartz, O-illite, H—kaolinite, M—montmorillonite, W—pyrite; L—anatase; N—other

3. Results and Discussion

Experiments were designed to test the expansion capacity of the #3 coals with different salinities after 8 h of immersion in fresh water and SDBS solutions with different concentrations. The results were analyzed at three aspects.

3.1 The expansion rate of coals under the SDBS solution with different concentrations within initial hour

The size and deformation rate of inflated coals should be illustrated by the change of instantaneous rate of coal samples when they are exposed to the external fluid [17]. Therefore, in order to reveal coal's swelling regular at the initial stage when filtrate permeats through coals, the expansion rate of coals under different concentrations of SDBS solutions within 1 h was studied. The results in Fig. 5 to Fig. 9, show the changes
of the expansion rates of the coal samples, all of which can be divided into three stages: rising, declining and fluctuating.

The expansion rate increased approximately linearly initially (about first 5 minutes). This is because the SDBS solution quickly permeated through coals contacting with the solution. Also the SDBS solution was weakly alkaline (pH≈9), while crystallographic montmorillonite of clay mineral in coal samples contained hydroxyls whose H^+ would disintegrate in the alkaline medium and absorb anions [18]. Meanwhile, the carbon-hydrogen bond of surfactant absorbed particles by van der Waals force. And the particles were some mass points with strong nonpolarity in montmorillonite layers. Then the negative charge of the montmorillonite layers increased due to the adsorption of negative ions, so the repulsive force between the layers also increased to expand the layer spacing [19]. Therefore, the expansion capacity of the coal samples rise sharply at the contact interface between the coal samples and the SDBS solutions. As a result, it is good to adjust the pH value of fracturing fluid in the process of preparation of anionic surfactant fracturing fluid according to the concentration of anionic surfactant. It can reduce the clay swelling at the moment when the fracturing fluid is exposed to the reservoirs.

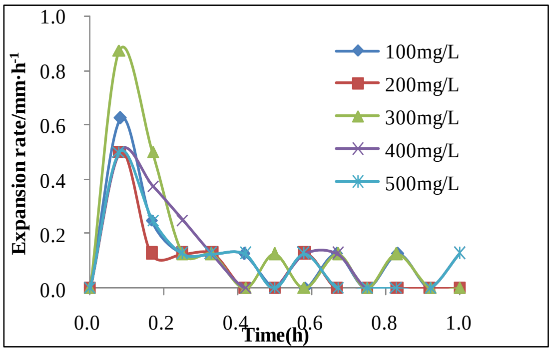


Fig. 5 *Expansion rate of coal samples at liquid of 1000mg/L salinity in different concentrations of SDBS solutions*

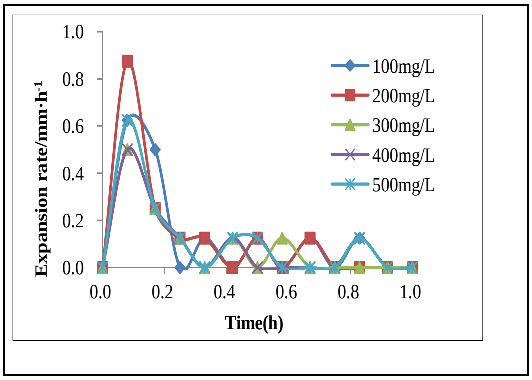


Fig. 6 *Expansion rate of coal samples at liquid of 2000mg/L salinity in different concentrations of SDBS solution*

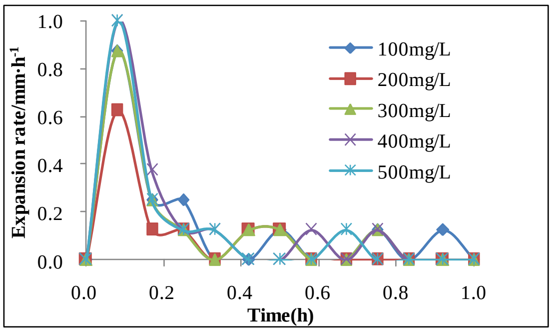


Fig. 7 *Expansion rate of coal samples at liquid of 3000mg/L salinity in different concentrations of SDBS solution*

At the same time, as the SDBS solutions permeated through the coals, Ca^{2+} of coals met the alkyl sulfonate of SDBS solutions and the Ca^{2+} tended to seep into the hydration layer of the surfactant polar head due to its small hydrated radius. Then the Ca^{2+} reacted with alkyl sulfonate (Fig. 10 (1)) and produced the positively charged complex-ion $Ca(RSO_3)^+$ which could absorb on the surface of negatively charged clay by electrostatic attraction (Fig. 9), such a reaction had proved by Yan [20-21]. Moreover, $Ca(RSO_3)^+$ displayed the partial bend conformation on the solid surface as shown in Fig. 9 and the directions of most lipophilic groups were towards the outside. This is helpful to make hydrophilic rocks turn to lipophilic groups and restrict the hydration expansion of clay. Therefore, the expansion rate reached a maximum after about 5 minutes, then down to the rapid decline stage and evolved to the stage of slow fluctuations.

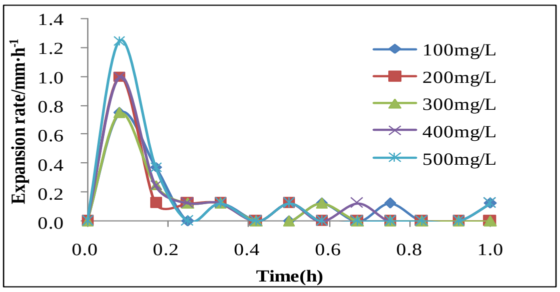


Fig. 8 *Expansion rate of coal samples at liquid of 4000mg/L salinity in different concentrations of SDBS solution*

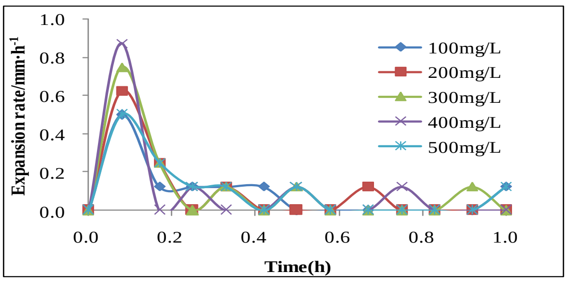
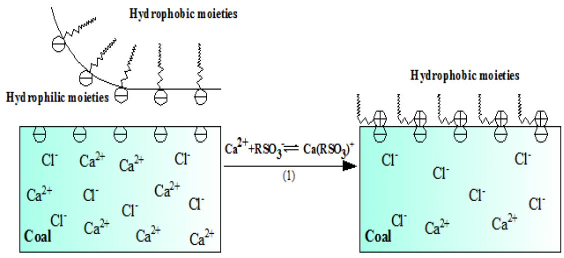


Fig. 9 *Expansion rate of coal samples at liquid of 5000mg/L salinity in different concentrations of SDBS solution*

3.2 Optimum concentration of anionic surfactant that was adapted to CBM reservoir with different salinities

The swelling capacity of the coal samples with different salinities in fresh water and SDBS solutions with different concentrations after 8 hours is shown in Fig. 11:



Note: R is the rest part except for the sulfonic group

Fig. 10 *Wettability effect of reaction between calcium ion and alkyl sulfonate on coal surface*

(1) The expansion capacity of untreated samples (samples without mineralization process) was stronger than those mineralization-treated samples, and it increased with the increase of SDBS concentrations. These are due to the electrostatic repulsion between the anionic surfactant SDBS and the clay's surface, which can maintain or even enhance the water wetness. In addition, the expansion capacity of treated samples immersed in fresh water was significantly greater than those immersed in SDBS solutions with different concentrations because the Ca^{2+} with smaller volume tended to interact with the hydrophilic head of SDBS when the Ca^{2+} in coals met the alkyl sulfonate of the nionic surfactant. And this can constrain the movement of the water molecules.

(2) The swelling capacity of coal samples immersed in fresh water and 100 mg/L SDBS solution become weaker with the increase of water salinity. The results suggest that the swelling capacity of the coal samples was mainly affected by water salinities of coal samples when the fresh water or SDBS solution with low concentration are used as fracturing fluids. It is known that the hydrated radius of calcium ions was small, the electrostatic attraction between the clay unit layer, Ca^{2+}, and clay unit layer was large, so the net potential energy between soil particles presented as an attraction [22-23]. Moreover, as the content of calcium ions increased, the attraction increased, and the water stability of samples would be better. As we can see from table 3, the specific gravity of clay minerals was large in coal samples, and in this test mineral ions mainly belonged to calcium ions. So the net potential energy between soil particles in coal samples presented as an attraction, and this attraction between the soil particles of coal samples with higher water salinities was larger

than those with lower water salinities, this meant that the swelling capacity of coal samples with higher water salinities was weaker than those with lower water salinities. In short, the swelling capacity of coal samples was mainly affected by formation salinities when samples were immersed in fresh water and SDBS solutions with low concentrations.

(3) With the increase of salinity, the expansion capacity of coal samples decreased, then increased, and afterwards declined slowly when samples were immersed in SDBS solution with high concentration. The reason was that when the samples' water salinity was relatively low, Ca^{2+} reacted with SDBS as the penetration of the SDBS solutions with high concentrations and their product $Ca(RSO_3)^+$ absorbed on the clay's surface in the form of monolayer adsorption and its lipophilic group was towards the outside, which led to the wettability reversal. In addition, adsorption between $Ca(RSO_3)^+$ and the clay surface turned from monolayer adsorption to double-layer adsorption when samples' water salinity increased, which could at first weaken then enhance the water wetness of clay surface .When the coal's water salinity reached a certain value, the concentration of Ca^{2+} and $(RSO_3)^-$ both increased up to an order of magnitude and the adsorption of generated $Ca(RSO_3)^+$ was saturated on the clay surface. In addition, the expansion capacity of coal samples declined somewhat because of the attraction between the Ca^{2+} and clay unit layer.

(4) Meanwhile, the minimum expansion capacity of coal samples with different salinities corresponded to different concentrations of SDBS solutions just because of the attraction between Ca^{2+} and clay unit layer as well as the complexation reaction between Ca^{2+} and SDBS. This was when the water salinities were 1000 mg/L, 2000 mg/L, 3000 mg/L, 4000 mg/L, 5000 mg/L respectively, and the corresponding optimal concentrations of SDBS solutions were 200 mg/L, 400 mg/L, 200 mg/L, 300 mg/L, 100 mg/L, respectively. The swell increment was 0.18 mm, 0.1 mm, 0.11 mm, 0.13 mm, 0.1mm respectively after 8 h and the inhibition of samples swelling reached a good level.

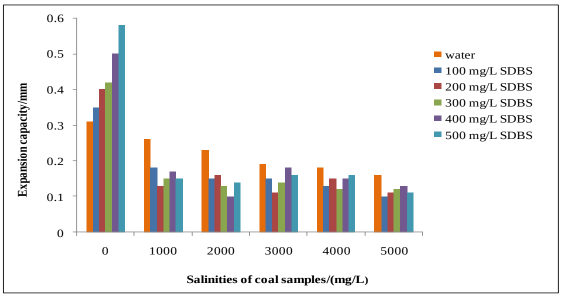


Fig. 11 *Impact of SDBS solution concentration and fresh water on the expansibility of coal samples*

3.3 The relationship between expansion rate, water salinities of coal samples and concentrations of SDBS solutions

In order to intuitively reflect the relationship between coal samples' swelling, its chemical composition and properties of the aqueous solution, the expansion ratio [24-25] was calculated for coal samples according to Eq. (1). Meanwhile, the water salinity 3000mg/L was used as the boundary for the higher and lower salinity and the fitted curves for concentrations of SDBS solutions to the coal samples' expansion ratio were obtained under the condition of relatively low and high water salinities. The results are presented in Fig. 12, and empirical expressions listed in Table 2.

$$\eta = \frac{\Delta H}{H} \times 100\% \tag{1}$$

Where η is the expansion ratio, ΔH is the swelling capacity of coal samples (mm), and H is the height of the original specimen.

According to Fig. 12, when water salinity was relatively low (≤3 g/L), the swelling capacity of coal samples decreased at first, and reached a minimum when the concentration of SDBS was 0.25 g/L, then increased as the increase of the concentration of SDBS. When the water salinity was relatively high (> 3 g/L), the swelling capacity of coal samples slowly increased as the increase of the concentration of SDBS.

Table 2 *Functions of expansion ratio and SDBS solution concentration*

Water salinity (g/L)	Curve equation
Low salinity (≤3 g/L)	$y = -1.888x^3 + 2.014x^2 - 0.633x + 0.152$
High salinity (>3 g/L)	$y = 0.291x^3 - 0.223x^2 + 0.074x + 0.077$

As mentioned above, the Ca^{2+} reacted with alkylbenzene sulfonate anion surfactant and produced a positively charged complex-ion $Ca(RSO_3)^+$. Then $Ca(RSO_3)^+$ absorbed on the clay surface, which played a role in inhibiting clay's hydration expansion. However, the system, in a certain concentration of inorganic salt, would continually produce complexed ions with the increase of anionic surfactant and the formed complex would dissolve [26] when endlessly adding the anionic surfactant. This would lead to the expansion of coal samples. Therefore, the concentration of Ca^{2+} of coal samples with low salinity was low and the effect of Ca^{2+} and coal samples' clay unit layer was not obvious, which meant the swelling capacity of coal samples was mainly affected by the complexation of Ca^{2+} and SDBS [20-21]. That was, with the increase of concentration of SDBS solution, coal samples' swelling capacity first basically decreased and then increased as the formation and dissolution of the complex. On the other hand, the swelling capacity was influenced by the coal samples' water salinities as well as the complexation between Ca^{2+} and SDBS when the coals' water salinity reached a certain value. It was found that the swelling capacity was mainly affected by Ca^{2+}-coal samples' clay unit layer under the low SDBS solution concentration. Then as the SDBS concentration increased, the complexation of Ca^{2+} and SDBS was enhanced and so the amount of Ca^{2+} decreased. At this point, the concentration of Ca^{2+} and $(RSO_3)^-$ both had increased by an order of magnitude and the adsorption of produced $Ca(RSO_3)^+$ achieved saturation on the clay surface. No matter how much $Ca(RSO_3)^+$ there was, it was still difficult to neutralize all the negative charge on the clay surface of the coal samples. So the function of clay's wettability being turned to oil wetting through producing positively charged complex by the Ca^{2+} was limited. Namely, the inhibitory effect in clay's hydration expansion was limited. Therefore, when the water salinity reached a certain value, the swelling capacity of coal samples slowly increased as the increase of the concentration of SDBS.

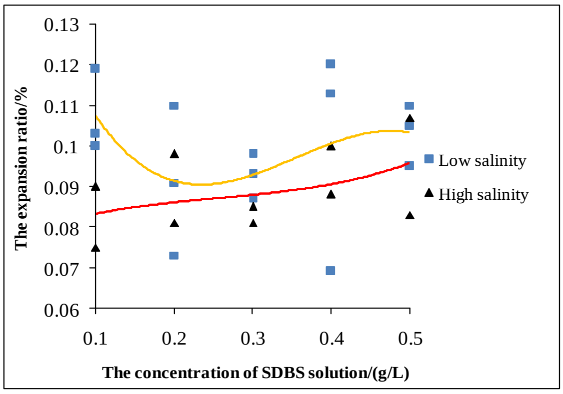


Fig. 12 *Expansion ratio at different SDBS solution concentrations for coal samples with different water salinities*

In the process of fracturing and production in CBM, it was recommended to use the relation in Table 2 to predict the expansion ratio of coals with water salinity and then adjust the formula of fracturing fluid to reduce the damage to the reservoir during the fracturing process.

4. Conclusions

By determining the swelling capacity of coal samples with different salinities immersed in water and SDBS solution with different concentrations, it was found that: as the water salinity increased, the swelling capacity of untreated samples, immersed in water or in SDBS solution with different concentrations, increased with the increase of the SDBS concentration, while the swelling capacity of treated samples, immersed in water or in SDBS solution with low concentration, became weaker. In addition, the minimum swelling capacity of coal samples with different salinities corresponded to different concentrations of SDBS solutions due to the attraction between Ca^{2+} and clay unit layer as well as the complexation reaction between Ca^{2+} and SDBS. Therefore, it is concluded that:

(1)Coal samples with water salinities had the inhibition effect in hydration swelling. This was mainly because of the wettability reversal effect of positively charged complex, produced by the complexation reaction between Ca^{2+} of coals and benzene sulfonate of SDBS, on coal surfaces.

(2)The effect of coals' water salinities on its hydration expansion was mainly embodied in the situation that the fracturing fluid was water or SDBS solution with low concentration. The higher the water salinity was, the stronger the attraction between Ca^{2+} and clay was, the smaller the effect of external filtrate on coals, and the better the relative stability of coal would be.

(3)The corresponding optimal concentrations of SDBS solutions were 200 mg/L, 400 mg/L, 200 mg/L, 300 mg/L, and 100 mg/L, respectively, when coals' water salinities were 1000 mg/L, 2000 mg/L, 3000 mg/L, 4000 mg/L, and 5000 mg/L, respectively.

(4)Determine the water salinities of coal reservoirs and use the relation $y = -1.888x^3 + 2.014x^2 - 0.633x + 0.152$ (salinity≤3000 mg/L) and $y = 0.291x^3 - 0.223x^2 + 0.074x + 0.077$ (salinity >3000 mg/L) to quantitatively predict the expansion ratio when SDBS solution with different concentrations permeate into coals, and then adjust the formula of fracturing fluid to reduce damage to the reservoirs during the fracturing process.

5. Acknowledgements

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