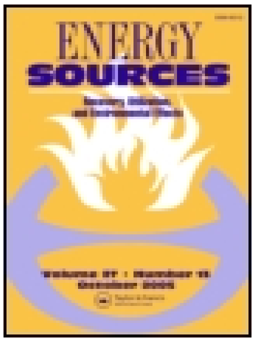

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Energy Sources, Part A: Recovery, Utilization, and Environmental Effects

ISSN: 1556-7036 (Print) 1556-7230 (Online) Journal homepage: https://www.tandfonline.com/loi/ueso20

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ENERGY SOURCES, PART A: RECOVERY, UTILIZATION, AND ENVIRONMENTAL EFFECTS
https://doi.org/10.1080/15567036.2019.1682723

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Experimental research on increasing effect of water injection in coal seam with chelating agent

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ABSTRACT

Coal seam water injection has a significant effect on the prevention and control of coal mine rock burst, and the pore characteristics of the coal seam is an important factor affecting the effect of coal seam water injection. With the deep mining of coal mine, the physical and mechanical properties of coal body change, the porosity of the coal seam decreases, and the injectability of the coal seam becomes worse. With the passage of time, the CO2 in the pores of the coal seam reacts with the easily precipitated ions to form precipitation sealing pores, which makes it more difficult to inject water. In order to obtain the reason why the water injection in coal seam becomes worse, the water injection additive with excellent performance is found, and a series of experiments on the impact tendency of coal body are carried out. The results show that the mine water used for coal seam water injection was liable to precipitate more ions, which could react with CO_2 dissolved in water to form precipitates, plug coal pore and reduce the fluidity of water in the coal seam, resulting in poor water injection effect. Adding chelating agent IDS in aqueous solution can effectively prevent precipitation formed by the combination of easily precipitated ions and carbonate ions in it. At the same time, the chelating agent IDS can decompose precipitated ions from the surface of coal particles, change from insoluble to soluble, promote pore development, increase the effect of water injection and the pore diameter of the coal, and reduce hydrophobic groups on the coal surface. IDS can reduce the impact tendency of coal, and it is an environmental protection additive that can increase permeability. It can provide reference for prevention and control of composite power disasters by coal seam water injection.

ARTICLE HISTORY
Received 29 May 2019
Revised 11 August 2019
Accepted 6 October 2019

KEYWORDS
Water injection in coal seam; chelating agent; effect of water injection; precipitable ions

Introduction

Rock burst is one of the most destructive dynamic disasters in coal mining. At present, there are more than 300 rock burst mines in China. Coal seam water injection technology can change the physical and mechanical properties of coal mass and the distribution of surrounding rock stress field, and reduce the dynamic disaster risk of working face. It is a commonly used mine power disaster prevention technology (AnsartR 2011; Cheng, Nie, and Zhou et al. 2012; Yin 2018; Zhang, Song, and Pan 2003; Zhang, Under Schultz, and Langhi et al. 2018; Zheng 2018; Zhou, Xia, and Wang et al. 2016). In the 1930s, prevention and control of coal and gas outburst by water injection in coal seams in former Soviet coal mines. In the early 1950s, coal seam water injection was once used to prevent and control rock burst. In the early 1960s, China has carried out coal seam water injection in Yangquan, Fushun, Tianchi and other mining areas to prevent coal and gas outburst, rock burst, etc. (Wu. 1982). The frequent occurrence of rock burst in the deep mining stage of thin coal seam of Xinxing mine,

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combined with the actual situation of the rock burst on the 41601 working face of the mine, the utility of coal seam water injection to prevent and control the rock burst of the thin coal seam has achieved remarkable results (Liu, Yuanping, and Zhang et al. 2013). However, as the mine enters deep mining, the coal stress will increase and the physical and mechanical properties of the coal change, especially the water injection impact of "high stress, strong adsorption and low permeability" coal seam will become worse, which cannot reduce the dynamic disaster risk of the coal seam.

Adding surfactant to the coal seam water injection can reduce the surface tension of water, enhance the water penetration ability, and enhance the water injection effect. Many scholars have done a lot of research on improving the effect of coal seam water injection with wetting agent. After the magnetization of the surfactant, the physicochemical properties and wettability of the coal changed (Ding et al., 2011). The adsorption capacity of surfactant CTAB to coal will increase with the increase of temperature, and the adsorption capability of oxidized modified coal is the highest (Marsalek, Pospisil, and Taraba 2011). The anion-nonionic surfactant mixture is more effective in reducing the apparent viscosity of the mixture than the cationic-nonionic surfactant mixture (Das, Dash, and Meher et al. 2013). The surfactant was mixed with the heat-collecting kerosene in a certain proportion to prepare the emulsion, which was used as a promoter to improve the hydrophobicity of unburned carbon (Zhou, Yan, and Wang et al. 2016). Surfactant chain length has little effect on the amount of cationic desorption (Li and Gallus 2007). The characteristics and water quality of coal and fine powder produced can reveal the root cause of coal permeability change (Guo, Hussain, and Cinar 2015). As the effective stress increased, the permeability decreased exponentially (Lu, Yang, and Ge et al. 2017). Pressure has a positive effect on water absorption, which increases with the increase of pressure, and the early water absorption rate of coal is faster (Chen, Jin, and Ma et al. 2011). Adding wetting agent can significantly improve the water injection impact of the coal seam. But for some deeply buried coal seams, due to the characteristics of "high stress and low permeability", the effect of adding wetting agent for water injection is still not good. The major performance is slow water injection speed, low water influx into a single hole, and after 3 to 5 days of water injection, the water injection volume is significantly reduced, or even not injected, which cannot meet the requirements of dynamic disaster prevention. Through on-site investigation and experiment in a large number of coal seam water injection field, it is found that the water used for coal seam water injection and the coal body contains a massive quantity of Ca^{2+}, Mg^{2+} and other ions, and the CO_2 adsorbed in the coal body will form a sure amount of CO_3^{2-}. CO_3^{2-} combines with Ca^{2+} and Mg^{2+} ions in the coal and in the injected water to form sediment. The sediment blocks the pores in the coal body, reducing the fluidity of the water in the coal seam, resulting in bad water injection effect. At present, the research on additives mostly focuses on increasing the wettability of coal. It is not considered whether the precipitates will block the water-conducting pores of the coal body and the influence of newly generated precipitates on the effect of water injection.

Because the anion of the chelating agent can form the complex with a high stable constant and ring structure with some metal ions, it is widely used in many fields. Heap leaching with EDDS is an effective way to reduce the content of heavy metals in contaminated soil (Hauser, Tandy, and Schulin et al. 2005). Chelating agents improve the catalytic performance of HDT catalyst (Mazoyer, Geantet, and Diehl et al. 2008). Al^{3+} can react with the chelating agent iminodisuccinic acid at 25℃ to produce a complex (Tolkacheva and Nikol'skii 2012), and consider that the coal layer incorporates a massive quantity of easily precipitated ions (Ca^{2+}, Mg^{2+}, $Fe^{2+/3+}$, etc.). Opening the secondary pores formed by these substances is the main means to increase the water injection effect of the coal seam in deep mining. Therefore, the chelating capacity of the chelating agent is used in this study to increase the effect of coal seam water injection (LijiSun.IDS 2016; Shi 2013; SU, Ling-Ling, and Hai-Long 2011; Wu 2015; Zhao 2016)

This paper carried out without additives, adding surfactants and contrast experiment under the condition of three kinds of the chelating agent, through the change of the mine water injection water convenient to precipitate after ion, reveals the mechanism of coal seam water injection gradually becomes poor, and further illustrates the chelating agent for coal pore effects. It provides a reference for studying the effect of additives on preventing and controlling rock burst by water injection in the coal seam.

Analysis of influencing factors of coal seam water injection effect

Effective analysis of deep coal seam water injection

The water injection effect of some coal seams in Pingdingshan mining area and Binchang mining area was investigated, and it was found that the coal seams in these deep coal mines were judged as injectable coal seams according to the *"MT/T 1023–2006 coal seam water injection comparison method"*. However, the water injection was carried out according to the existing water injection parameters, it is found that water injection can still inject part of water in the initial stage, but the daily injection amount of water injection hole is low, and the daily injection water quantity of water injection hole decreases gradually with the passage of time, and it is basically impossible to inject water into the hole after a few days of water injection. Through a lot of investigation and tests, it is found that the water injected by coal mine is of excessive hardness and easy to precipitate more ions. However, when CO_2 adsorbed in pores is dissolved in water, it reacts with precipitated ions to form precipitation sealing pores or reduce pore diameter to reduce the mobility of water and reduce the effect of water injection. This is an important factor causing the water injection effect to deteriorate. For example, by sampling and analyzing the water used for water injection in a coal seam in Pingdingshan mining area, it is determined that the content of important precipitable ions in the water is relatively high, and the results are shown in Table 1.

Change of coal porosity after hard water action

The standard coal sample was taken, the initial porosity of the coal sample was measured, and the coal sample was dried. CO_2 was absorbed into coal sample by triaxial adsorption device, and then $CO_2$was adsorbed in pores, then dripped slowly into hard water, repeated several times and dried, and the porosity of coal after action was measured.

It is found that the injected water has an excessive hardness from Table 1, and Table 2 indicates the sealing effect of the pores after the formation of precipitation. The water with high hardness has a great effect on pore sealing. According to the experimental results, part of the reason for the poor water injection effect of the coal seam is that the CO2 adsorbed in the pores of the coal dissolves in the solution to form carbonate ions, which combine with the easily precipitated ions in the coal and the injected water to form a precipitate. The generated sediment blocks the original pores of the coal body, reduces the fluidity of the water in the coal seam, and the water injection effect becomes worse and worse.

Selection of additives

This paper analyzes the reasons for the poor effect of water injection in existing mines, and it is necessary to add additives to the water injection to solve the problem of pore sealing and open the pores to increase the effect of water injection. Because the chelating agent can form a stable constant

Table 1. Precipitable ion content in water for coal seam water injection.

Precipitable ion $\mu g{\cdot}mL^{-1}$	Ca^{2+}	Mg^{2+}	Fe^{2+}/Fe^{3+}
The water injection	78.8	17.5	1.85
Tap water	50.5	10.4	0.66

Table 2. Changes of porosity before and after coal sample treatment.

	Initial porosity *n*%	Porosity after treatment *n*%
The water injection	4.3	2.5
Tap water	4.1	3.6

with the easily precipitated ions in the coal sample and has a ring structure complex, it has strong stability in a wide range of pH. Through the dissolution of soluble minerals, the connectivity of pores in the coal body is improved, the pores in the coal body are increased, and the effect of water injection is realized. At the same time, the combination of easily precipitated ions and carbonate ions to form precipitation and re-plug pores can be prevented, so chelating agents can be applied in coal seam water injection. Tetrasodium iminodisuccinate is called IDS for short. Due to its green environmental protection, low price, good degradability, surface activity, strong chelating ability, and other advantages, IDS was selected as the representative of the chelating experiment in this paper. In the field of water injection in coal seam prevention and control of impact earth pressure, no research and report have been made.

The IDS chosen in this paper is the structural method of analytically pure, structural formula of chelating agent IDS, as shown in Figure 1. In order to verify that IDS can enhance the performance of coal seam water injection, the precipitation ion content (taking $^{Ca2+}$ as an example) in the water injected by IDS complexation was experimentally studied. Two 100 mL volumetric flasks have been stuffed with 200 mL of the injected water, one of which used to be IDS powder, and then the same amount of CO_3^{2-} used to be introduced to the two solutions, respectively, to decide the remaining Ca^{2+} convenient to precipitate content. The test results are shown in Table 3.

According to the data in Table 3, the Ca^{2+} content in the solution added with IDS is 35 times higher than that in the solution not added with IDS. The anion of IDS forms a stable complex with Ca^{2+}, which is very effective in preventing the combination of Ca^{2+} and CO_3^{2-} to form $CaCO_3$ precipitate, preventing sedimentation and sealing coal pores and reducing the water injection effect.

The additives commonly used in coal seam water injection, sodium dodecylbenzenesulfonate (SDBS) and sodium dodecyl sulfate (SDS), were selected to compare the change rules of Ca^{2+}content material in the solution through experiments. The injected water was put into a 100 mL volumetric flask, sodium dodecylbenzenesulfonate, sodium dodecyl sulfate, and IDS had been added, and then CO_2 gas was once slowly injected for 24 h. The content of Ca^{2+}in the solution was determined by ICP. The results are shown in Table 4.

As can be seen from the statistics in Table 1, the Ca^{2+} content in the injected water is 78.8 $g{\cdot}ml^{-1}$, while the Ca^{2+} content in the authentic filtrate is decreased by almost 1/2 after the injection of CO_2, and rarely diminished after the addition of IDS. At the same time, in contrast with other wetting

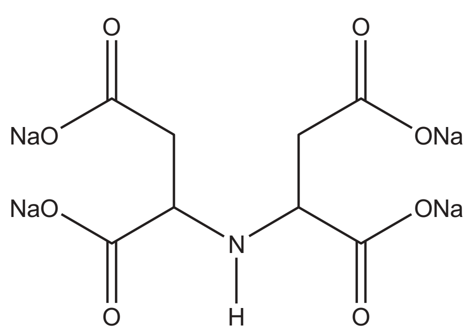


Figure 1. The structural formula of chelating agent IDS.

Table 3. Changes of Ca^{2+} in solution after the addition of CO_3^{2-}

Solution type	Water injection	Adding the IDS
Ca^{2+} content ($\mu g{\cdot}mL^{-1}$)	1.85	66.2

Table 4. Ca^{2+} content in different solutions passing through CO_2.

Solution type	Water injection	Adding the SDBS	Adding the SDS	Adding the IDS
Ca^{2+} content ($\mu g{\cdot}mL^{-1}$)	36.0	12.6	20.4	70.4

agents, it can be seen that IDS can correctly prevent the precipitated ions in the injected water from combining with carbonate ions to form precipitation.

Analysis of chemical mechanisms of IDS for precipitable ions in coal

Effect of IDS concentration on leaching rate of precipitable ions

Grinding the coal sample and passing through 1 mm sieve, 10 g of coal powder was weighed and placed in a jar, and then the IDS solutions with awareness gradient of 0, 500, 1000, 1500, 2000, 2500, 3000 and 3500 mg/L had been prepared with ultra-pure water. Pour different concentrations of IDS answer into jars containing pulverized coal and soak for 9 days. After centrifugation and filtration of the supernatant, ICP was used to decide the effect of IDS solutions with unique concentrations on the precipitable ions in coal, as proven in Figure 2, and the Ca^{2+}, Mg^{2+}, Fe^{2+}/Fe^{3+} ion concentrations in IDS solutions have been fitted.

The standard curve equation of Ca^{2+} ion concentration is

$$y = 2E + 06x - 413434$$

The square value of the correlation coefficients

$$R^2 = 0.9996$$

The standard curve equation of Mg^{2+} ion concentration is

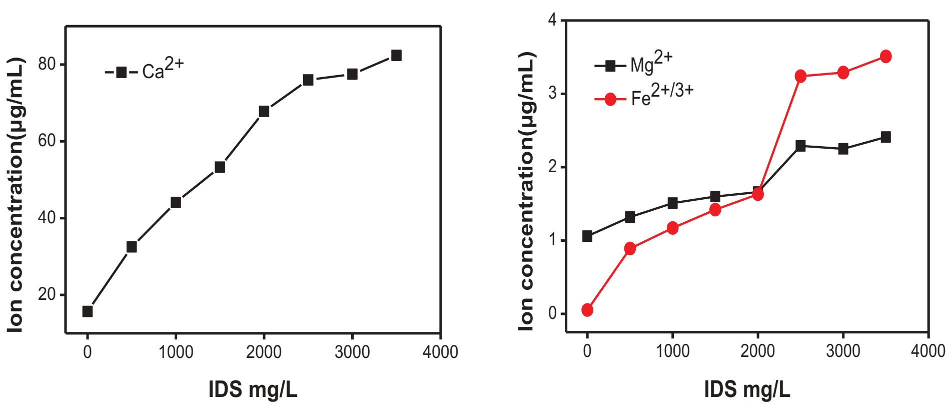
$$y = 374885x + 2641.2$$

The square value of the correlation coefficients

$$R^2 = 0.9996$$

The standard curve equation of Fe^{2+}/Fe^{3+} ion concentration is

$$y = 6061.2x + 36.2$$



a) Ca^{2+} leaching rate of coal after the action IDS b) leaching rates of Mg^{2+} and Fe^{2+}/Fe^{3+} in coal after the action of IDS

Figure 2. Easy to precipitate ion leaching rate after soaking with different concentrations of IDS solution.

The square value of the correlation coefficients

$$R^2 = 0.9996$$

Figure 2 indicates that with the increase of IDS solution concentration, the leaching rate of precipitable ions presents an increasing trend, especially Ca^{2+} ions. Compared with blank samples, the leaching rate of Ca^{2+} after the action of IDS answer is 5.25 times higher than that of ultra-pure water. The Ca^{2+} leaching rate was significantly increased when the IDS answer awareness was 0 to 2500 mg/L. When IDS solution concentration is between 2500 and 3500 mg/L, the increasing trend of ion leaching rate slows down.

After the action of IDS solution, the leaching rate of Mg^{2+} indicates an upward trend, but the average change is no longer very great, and when the concentration reaches 2500 mg/L or above, the content of Mg^{2+} is nearly unchanged, probably because IDS has completely chelated the Mg^{2+} in the coal powder.

After immersion in ultra-pure water, the concentration of Fe^{2+}/Fe^{3+} is only 0.052 μg/mL, while the leaching rate of Fe^{2+}/Fe^{3+} increases linearly after the action of IDS solution, and is 67.5 times higher than the blank sample at the highest. According to Figure 2, when the concentration of IDS reaches 2500 mg/L or above, the leaching rate of Fe^{2+}/Fe^{3+} has little alternate and reaches equilibrium.

Effect of immersion time of IDS solution on leaching rate of precipitable ions

The concentration of IDS was 2000 mg/L, the solid–liquid ratio was 10 g:100 mL, and the immersion time was 1d, 3d, 5d, 7d, and 9d. After soaking, solid–liquid separation was carried out, and then ICP was used to notice the impact of IDS solution at unique soaking instances on precipitating ions in coal, as shown in Figure 3.

As can be seen from Figure 3, the leaching rate of Ca^{2+} is very small with the change of soaking time, while the leaching rate of Fe^{2+}/Fe^{3+}, Mg^{2+} will increase first and then decreases with the change of soaking time. The reason may be that there are differences in the content of easily precipitated ions in coal samples and the change of temperature may have some influence on the chelating properties of chelating agents, resulting in minor differences.

The influence of solid–liquid ratio on the leaching rate of precipitated ions

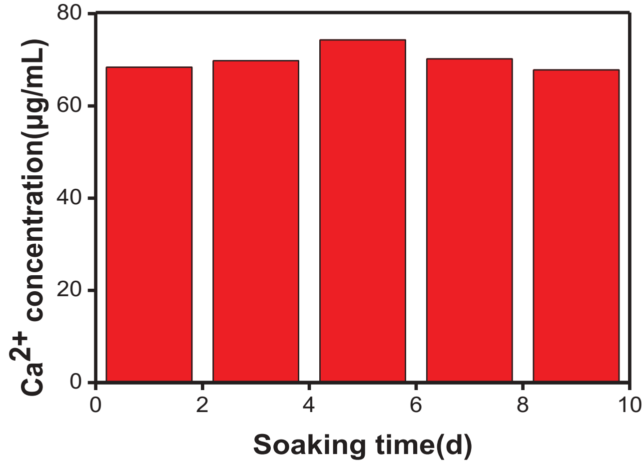
Selection of IDS concentration of 2000 mg/L, the soaking time is 9 days, and the 10 g:100 mL mass ratio is 1:10. The selected solid–liquid ratio is 1:5, 1:10, 1:20. Under different solid–liquid ratio conditions, the easily precipitated ion leaching rate can be obtained, as shown in Figure 4.

It can be seen from Figure 4 that the solid–liquid ratio of the IDS solution and the coal sample has little effect on the easy-precipitation ion leaching rate, especially the calcium ion has almost no difference. When the solid–liquid ratio of IDS solution to coal sample is 1:5, the leaching rate of Fe^{2+}/Fe^{3+} and Mg^{2+} is higher than that of other ratios. It can be seen from Figure 2–4 that the IDS solution has obvious effect on the easily precipitated ions in the coal. The easily precipitated ions and the coordination atoms in the IDS form a complex with a cyclic structure, which can make the calcium, magnesium and iron plasmas from the coal surface. The solution is decomposed, from insoluble to soluble, and separated from the coal. At the same time, it can prevent the CO_2 adsorbed in the coal layer from being dissolved in water and chemically react with water to form carbonic acid. If IDS is not added, the CO_2 in the coal will dissolve in water to form carbonic acid, further ionize to form H^+, HCO_3^-, and HCO_3^- continues to ionize to form H+, CO_3^{2-}. The chemical equation is as follows:

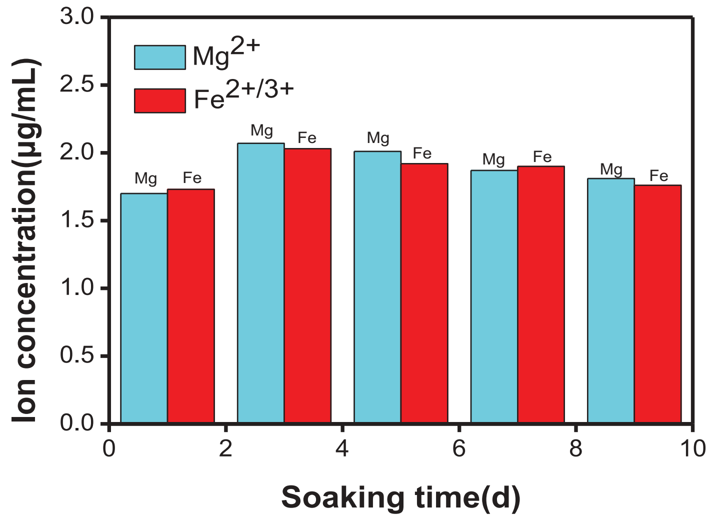
$$CO_2 + H_2O \rightarrow H_2CO_3$$

$$H_2CO_3 \rightarrow H^+ + HCO_3^-$$

$$HCO_3^- \rightarrow H^+ + CO_3^{2-}$$



a) changes in Ca^{2+} leaching rate of coal indifferent soaking time in coal



b) changes in the leaching rates of Mg^{2+} and Fe^{2+}/Fe^{3+}at different soaking times

Figure 3. Effect of time on easily precipitated ions in IDS chelated coal.

CO_3^{2-} combines with easily precipitated ions such as Ca^{2+} to form $CaCO_3$ precipitation to replug the pores and weaken the effect of water injection.

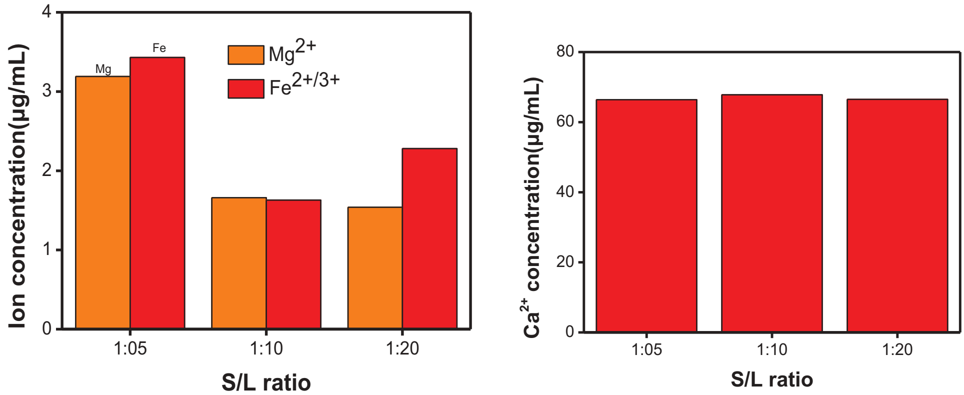
Porosity change of coal sample after soaking in IDS solution

The changes of porosity of coal samples treated with chelating agent IDS are shown in Table 5.

According to the data in the table, it can be seen that the porosity of the coal sample after the motion of IDS chelating agent is nearly 4 times greater than the initial porosity, and the porosity of the coal sample after soaking in ultra-pure water is about 2 times greater than the preliminary porosity. It can be seen the IDS solution after coal pattern porosity extensively open, in the main due to the fact of the chelating agent below the addition of wetting marketers and minerals in the coal pattern to chelation winding down surface of minerals from coal particles in answer by the insoluble

Table 5. Changes in the porosity of coal sample after chelating agent action.

	Initial porosity *n*/%	Post-treatment porosity *n*/%
Immerse the IDS	2.1	8.3
Ultrapure water	2.0	3.5



a) influence of solid-liquid ratio on the leaching rates of Mg^{2+} and Fe^{2+}/Fe^{3+} in coal leaching

b) influence of solid-liquid ratio on Ca^{2+}rate of coal

Figure 4. Effect of solid–liquid ratio on precipitable ions in IDS chelated coal.

country into soluble state, to open the secondary porosity enlarge liquidity in the water, extend water injection effect.

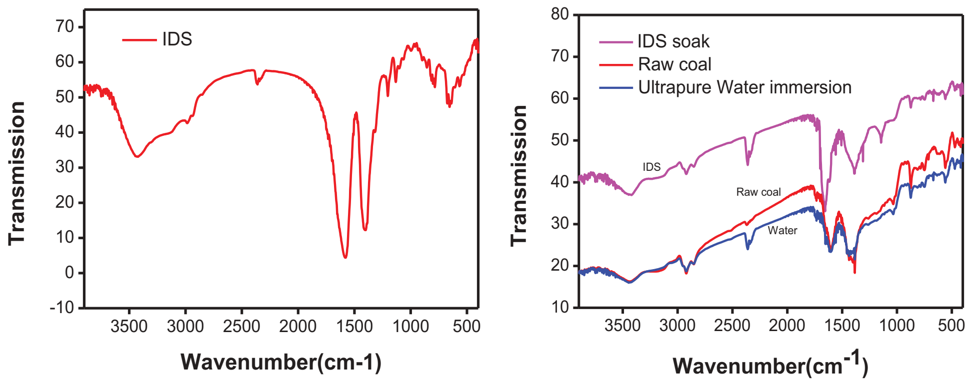
Change laws of functional groups in coal samples before and after soaking

The surface of the coal body mainly contains a hydrophilic group and a hydrophobic group, and the content of the group affects the wettability of the coal. The variation of the hydrophilic and hydrophobic functional groups of coal after the action of chelating agent IDS was determined by Fourier transform infrared spectrometer. The soaking time was 9 days. The coal samples were compared with unsoaked, ultrapure water soaked and IDS solution soaked (IDS concentration 2000 mg/L), and the infrared spectrum of analytical reagent IDS was analyzed. The test results are shown in Figure 5.

By comparing the infrared spectra of the unsoaked and IDS soaked coal samples, it can be viewed that there is a huge difference in the absorption peaks between 1700–1600 cm^{-1} and 1200-1100 cm^{-1}, and the hydrophilic practical businesses are in this region. However, there is a big difference in the absorption height electricity between the unsoaked and IDS soaked coal samples in the stages of 1590–1370 cm^{-1}, 910–700 cm^{-1} and 2999–2800 cm^{-1}, and the foremost purposeful companies are hydrophobic. In conclusion, after soaking the coal samples with IDS, the hydrophilic purposeful businesses on the floor of the coal amplify while the hydrophobic functional agencies decrease. IDS solution also helps increase the wettability of coal surfaces.

The impact of IDS on coal mine bursting liability

Coal sample dynamic failure time, elastic energy index, impact energy index, and uniaxial compressive strength are indicators of coal seam bursting liability. When these bursting liability warning signs are large than the integral value, it is possible to have rock burst.



a)IDS infrared spectrum

b) infrared spectrum after the action of IDS

Figure 5. Infrared spectra of coal samples and IDS.

Sample collection, processing, and testing are carried out in strict accordance with the provisions of *GB/T 23561–2009, "methods for the determination of physical and mechanical properties of coal and rocks"*, the national standard of the People's Republic of China. Coal impact tendency appraisal strictly comply with national standards of the People's *Republic of China the impact ground pressure measurement, monitoring, and prevention method part 2: the impact of coal tendentiousness and the index and classification method of GB/T 25217.2 2010*, and with reference to the international society for rock mechanics laboratory and field test standardization commission on *the rock mechanics test solution*.

According to Figure 6 and Table 6, it can be considered that the dynamic failure time of coal increases by more than 4 times, both the elastic energy index and the shock energy index dropped significantly, and uniaxial compressive strength decreased by more than 4 times. It can be seen that IDS has a very significant effect on reducing impact propensity. This is because IDS releases some mineral ions in the coal, reducing the density and strength of the coal, which is the hardest part of the coal, thus affecting the bursting liability.

Conclusion and prospect

When the coal seam water injection method is adopted to prevent and control dynamic disasters, the CO_2 in the coal seam will be dissolved in the water solution, and the shaped CO_3^{2-} will combine with the easily precipitated ions dissolved in the water injection and the coal to form precipitated substances, which will be deposited in the pores of the coal physique and seal the pore water passage, resulting in negative water injection effect.

Through experimental comparison, it is found that the content of Ca^{2+} in the original filtrate without adding IDS hardly ever decreases after the CO_2 is injected, while the content of Ca^{2+} in the original filtrate with including IDS reduces by extra than half after the CO_2 is injected. The chelating agent IDS can prevent the combination of precipitated ions and carbonate ions from forming precipitates and seal the pores.

The chelating agent IDS solution can interact with each mineral, Ca^{2+} was increased by 5.25 times, Fe^{2+}/Fe^{3+}increased by 67.5 times compared with that of ultra-pure water immersion with chelating agent IDS. The effect of chelating agent IDS solution on Mg^{2+} leaching charge was once notably weak. The time of soaking coal sample and solid–liquid ratio have little influence on the leaching rate of precipitating ions, while the concentration of IDS has an outstanding influence on the leaching rate of precipitating ions.

After the coal physique is soaked by the chelating agent IDS solution, the hydrophilic group content material on the coal surface increases, while the hydrophobic group content decreases, and the change of organizations increases the wettability of the coal.

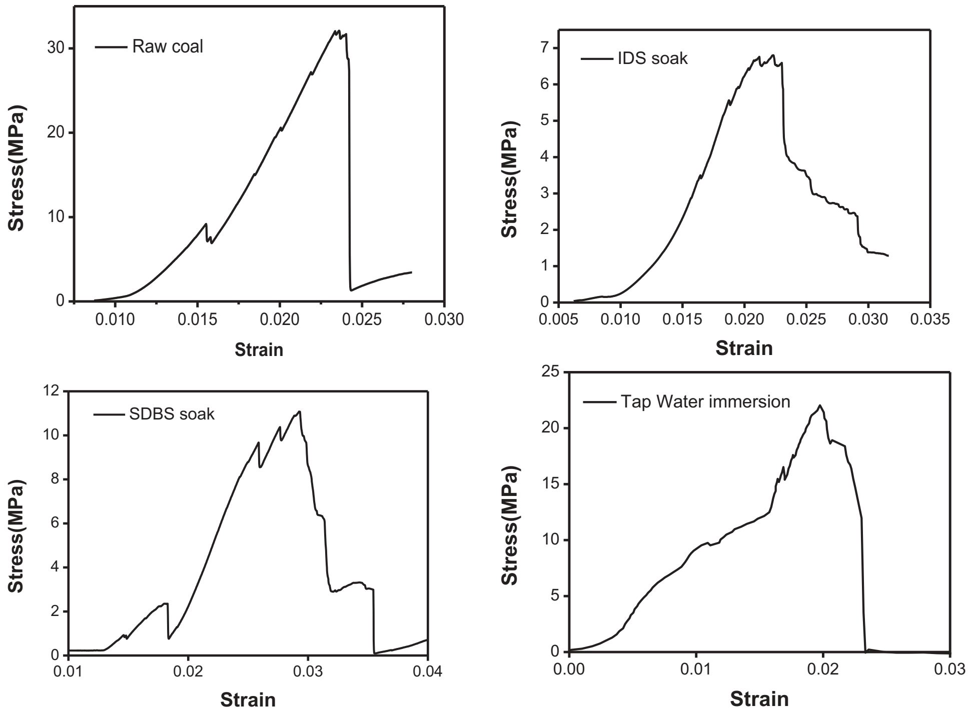


Figure 6. Change of coal impact tendency after soaking.

Table 6. Specimen dimension table.

	Dynamic failure time DT/ms	Impact energy index KE	Elastic energy index WET	Uniaxial compressive strength Rc/MPa
Raw coal	324	4.85	2.85	32.1
Tap water	509	3.54	2.01	21.6
SDBS	727	3.12	1.62	11.1
IDS	1305	1.84	0.96	6.8

Minerals are the hardest part of coal. After the role of IDS, minerals are released from the surface of coal, which can also reduce the density and strength of coal and thus reduce the impact tendency of coal.

Acknowledgments

The authors gratefully acknowledge Pingdingshan No.11 Mine for the financial resources provided, Liaoning University for the performed instrumental analysis. This paper was supported by the State Key Research Development Program of China (Grant No. 2017YFC0804208).

Funding

This work was supported by the National Key Research and Development Project [2017YFC0804208],The National Natural Science Foundation of China[51904141], the Educational Commission of Liaoning Province of China [LQN201705].

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