

Experimental Investigation of the Basic Characteristics and Wettability of Oil Shale Dust

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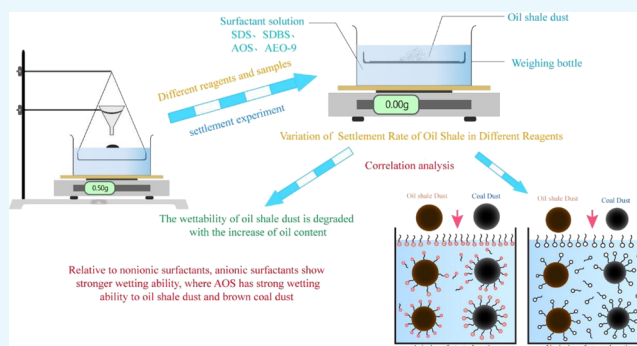
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ABSTRACT: To investigate the influence of basic characteristics of oil shale dust on wet dust removal, oil shales (Longkou oil shales Y_1 , Y_2S_2 , and Y_2S_1 and Fushun oil shale) with different oil contents, brown coal (M_1), and bituminous coal (M) were selected from a typical mining area in China to test their physiochemical parameters. Their proximate component, chemical structures, surface morphology, and mineral contents were determined. The sedimentation experiments of oil shale dust and coal dust were implemented using three anionic surfactants (AOS, SDS, and SDBS) and a nonionic surfactant (AEO-9), and the wettability of oil shale dust was analyzed and compared with that of coal dust. The experimental results indicate that the moisture content, volatile content, fixed carbon content, and content of oxygen-containing functional groups of oil shale are higher than those of the coal sample; otherwise, contents of ash, SiO_2 , aliphatic hydrocarbon, and aromatic hydrocarbon are lower. It can be found that oil shale has semiclosed pores, poor connectivity, a small pore size, a large specific surface area, and a rougher surface, which will lead to poorer wettability of oil shale. The wettability of oil shale can be improved by adding surfactants but is still weaker than that of the coal samples. Anionic surfactants are better than nonionic surfactants in improving the wetting performance. Among them, AOS shows strong wetting ability to oil shale dust. The research results of this paper have an important practical significance for analyzing the wettability of oil shale and controlling oil shale dust.



INTRODUCTION

Oil shale is an important energy resource and is only second to coal reserves in the world in terms of the equivalent calorific value.^{1–4} In fact, under the background of increasing energy consumption and reduced petroleum supply, oil shale has been identified as a recognized important alternative energy in the future by virtue of abundant reserves and high exploitability.^{5–8} During the exploitation process of oil shale, traditional underground mechanical oil shale exploitation is an important mode. With special physiochemical properties, oil shale has problems like high dust content, low porosity, and difficult permeation in the mechanical exploration process. The dust removal efficiency of oil shale is seriously restricted due to its poor wetting effect.^{9–13} Therefore, the wettability of oil shale dust is a key factor influencing the spray dust removal effect. Hence, studying the wettability of oil shale dust is of great importance to improving dust suppression efficiency.^{14–16}

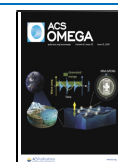
At present, there are a few research studies on the wettability of oil shale dust, and many domestic scholars have investigated the wettability of coal dust and the results show that surfactants can improve the wettability of coal dust.^{17–20} Adding a proper amount of surfactant in water can effectively reduce the surface tension of the solution and form an adsorbed layer structure on

the dust surface so as to change the dust wettability and make it more hydrophilic.^{21,22} The wetting effect of dust is associated with its physiochemical characteristics and type and the concentration and surface tension of surfactants. Wang *et al.*²³ determined the contact angles and Fourier transform infrared spectra of five coal samples. The results show that benzene ring, benzene ring-containing aromatic hydrocarbon, and aliphatic hydrocarbons containing methyl, methylene, and so on are all hydrophobic and oxygen-containing functional groups represented by hydroxyl and carboxyl are hydrophilic. Li *et al.*²⁴ studied relationships of granularity and fractal dimension of three different coal samples with wettability. The results indicate that as the grain size of coal dust increases, the microstructure becomes more complicated with degraded wettability. Wang *et al.*^{25,26} tested the abilities of different surfactants to wet coal

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samples via the sedimentation experiment based on different physiochemical characteristics of the coal samples. According to the results, the wetting ability of the anionic surfactant is stronger than the nonionic surfactant, and the wetting ability varies from coal sample to coal sample. Therefore, a systematic study of physiochemical characteristics of six types of oil shales with different oil contents and coal samples was carried out in this paper, and meanwhile, the influences of surfactants on their wettability were determined. The study of basic characteristics and wettability of oil shale dust provides a theoretical basis for prevention and control of oil shale dust.

RESULTS AND DISCUSSION

Influence of Proximate Components on the Wettability of Oil Shale Dust and Coal Dust. The results of

Table 1. Results of Proximate Analysis

sample	proximate analysis (wt %)			
	M_{ad}	A_{ad}	V_{ad}	FC_{ad}
F	2.86	71.05	23.75	2.34
Y_2S_2	3.89	72.89	22.2	1.02
Y_2S_1	2.68	70.07	25.11	2.14
Y_1	11.34	20.28	42.68	25.7
M_1	16.9	7.27	33.03	42.8
M	15.57	7.4	35.22	41.81

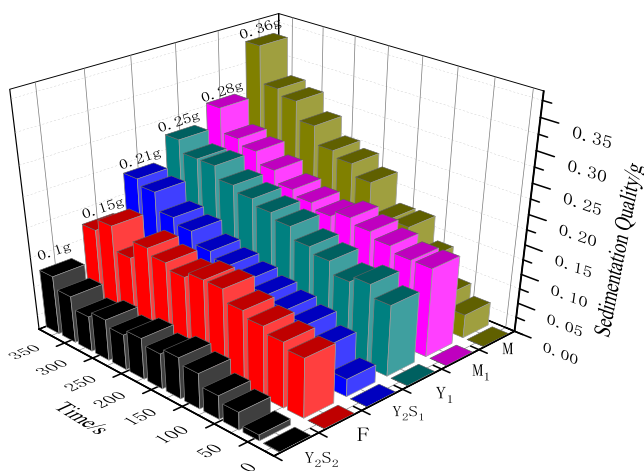


Figure 1. Sedimentation experimental results of oil shale dust and coal dust.

proximate analysis are shown in Table 1. The proximate analysis aimed to acquire proximate compositions of moisture, ash content, volatile component, and fixed carbon content of oil shale and coal samples under specific conditions.²⁷ It could be seen from Table 1 that in comparison with bituminous coal M and brown coal M_1 , oil shale was characterized by low moisture, low volatile content, low fixed carbon content, and high ash content. The moisture content of bituminous coal was four times that of oil shale Y_2S_2 , and the ash content of oil shale Y_2S_2 was the highest, reaching 72.89% and being 9.85 times that of bituminous coal.

By comparing oil shales in different areas, the change laws of four indexes of Longkou oil shales were the same as those of Fushun oil shale, both belonging to highly ashy substances. The ash content, volatile content, and moisture content in Fushun oil shale were all lower than those of Longkou Y_2S_2 , and its fixed

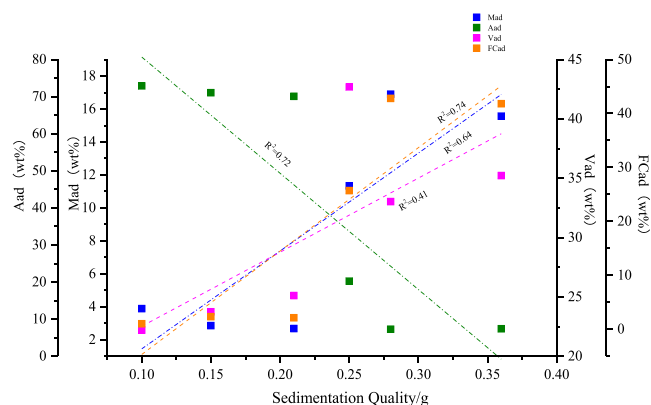


Figure 2. Correlation analysis between proximate components and sedimentation mass.

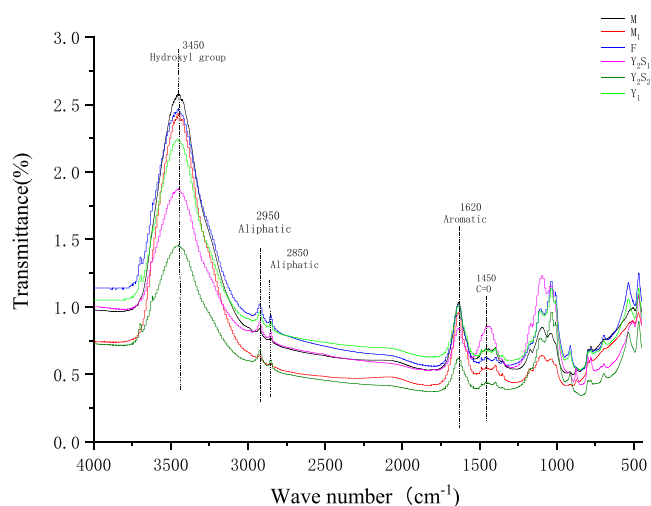


Figure 3. FTIR spectrograms of oil shale dust and coal dust.

carbon content was twice that of Y_2S_2 . For oil shales with different oil contents in the same area, the ash content in oil shale increased with the oil content, but the contents of moisture, volatiles, and fixed carbon presented a declining tendency. As the oil content increased, the ash content rose from 20.28 to 72.89%, indicating that the mineral content would increase with the oil content, and the volatile content declined from 42.68 to 22.2%, manifesting that the volatile content decreased with the increase in oil content.

The sedimentation mass changes of oil shale dust and coal dust in distilled water are presented in Figure 1. In the sedimentation experiment, the greater the sedimentation velocity, the better the wettability.²⁸ By comparison, it is found that M had the best wettability in the six samples and the wettability of M_1 was only second to M, but the wettability of oil shale was weakened with the increase in oil content and that of Fushun oil shale was between those of Longkou Y_2S_2 and Y_2S_1 ; thus, their wettability was sorted as follows: $Y_2S_2 < F < Y_2S_1 < Y_1 < M_1 < M$. Origin9.1 software was used to do linear fitting of moisture, ash content, and volatile matter of the samples as well as sedimentation mass of coal samples as shown in Figure 2. The wettability of oil shale dust and coal dust had positive correlations with moisture ($R^2 = 0.64$) and fixed carbon ($R^2 = 0.74$) while having a negative correlation with ash content ($R^2 = 0.72$), and its linear correlation with volatiles was weak, where the correlation coefficient was only $R^2 = 0.41$, meaning that the

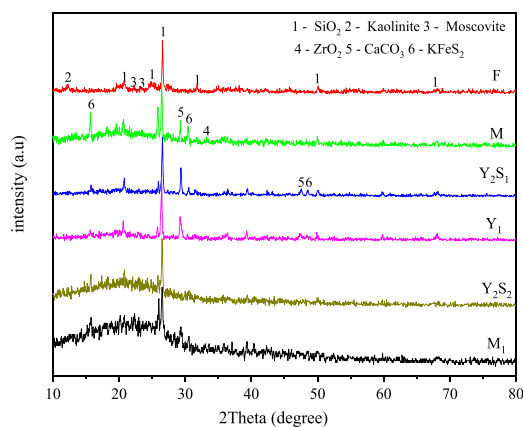
Table 2. Characteristics and Attribution of FTIR Absorption Peaks of Oil Shale and the Coal Sample

type of functional group	number	peak position/ cm ⁻¹	functional group	attribute
hydroxyl	A	3500–3200	–OH	Intermolecular hydrogen bonding of the phenolic hydroxyl group, alcoholic hydroxyl group, or amino
aliphatic hydrocarbon	B	2975–2915	–CH ₂ –CH ₃	Asymmetric stretching vibration of methyl and methylene
	C	2875–2850	–CH ₂ –CH ₃	Symmetric stretching vibration of methyl and methylene
	D	2950	–CH ₃	Symmetric stretching vibration of methyl
aromatic hydrocarbon	E	3053–3030	–CH	Stretching vibration of aromatic hydrocarbon
	F	1630–1600	–C=C–	–C=C– stretching vibration of the conjugated double bond
	J	1348	–CH ₃ –CH ₂	Deformation vibration of methyl and methylene of aliphatic groups
	H	867	H	Out-of-plane deformation vibration of two adjacent hydrogen atoms on the same aromatic nuclei in aromatic hydrocarbon
	I	750	H	Out-of-plane deformation movement of four and five adjacent hydrogen atoms on the same aromatic nuclei in aromatic hydrocarbon
oxygen-containing functional group	J	1710–1700 1800–1300	C=O	Stretching vibration of aldehyde, ketone, and acid carbonyl
	L	1330–1100	C–O	Stretching vibrations of phenol, alcohol, ether, and ester
	M	1060–1170	Si–O	Stretching vibration of the Si–O bond

Table 3. Analysis of Minerals Contained in Oil Shale and Coal Samples^a

sample mineral	SiO ₂	CaCO ₃	KFeS ₂	ZrO ₂	kaolinite	muscovite
F	69.5	×	×	×	2.7	27.8
Y ₂ S ₂	91.3	2.1	6.6	×	×	×
Y ₁	75.7	16.6	8.2	×	×	×
Y ₂ S ₁	75.2	16.6	8.2	×	×	×
M ₁	70	16.6	10	3.4	×	×
M	60.5	9.9	18.6	2	×	×

^aNote: × means not detected.

**Figure 4.** X-ray diffractograms of oil shale and coal samples.

volatile content was not the primary factor of the wettability of oil shale dust. Moisture in oil shale contained free water and bound water, and the water obtained through the proximate analysis was mainly free water in oil shale. Free water is water in internal pores, which is adsorbed on the outside surface of oil shale to combine with oil shale in a physical state. In the proximate analysis, higher moisture content indicated more developed internal pores.

Influence of Chemical Structure on the Wettability of Oil Shale Dust and Coal Dust. The wettability of oil shale dust is not only related to its composition but also to its chemical structure. An FTIR spectrum can accurately characterize functional groups, positions, and contents of organic macromolecular structures and it is one of important means of

investigating organic macromolecular structures.^{27,28} FTIR spectral analysis and characterization of the chemical structure and functional groups of oil shale dust were carried out, where the FTIR spectrograms are shown in Figure 3 and absorption peaks of the functional groups are shown in Table 2.

It could be seen from Figure 3 and Table 2 that bituminous coal and brown coal contained many oxygen-containing functional groups, while the content of aliphatic hydrocarbons was relatively small, and the contents of aliphatic hydrocarbons and aromatic hydrocarbons in oil shales were large. Peak positions in oil shales in different areas were basically identical but with different peak intensities. In the 2950–2850 cm⁻¹ region, it was the stretching vibration of aliphatic hydrocarbons to different degrees, and the law was presented as follows: F > Y₂S₂ > Y₂S₁ > Y₁ > M₁ > M. It could be known that the content of aliphatic hydrocarbons in Fushun oil shale was higher than those of Longkou oil shales and the control coal samples. Meanwhile, as the oil content increased, the content of aliphatic hydrocarbon in Longkou oil shale Y₂S₂ was at the maximum; At 1620–1590 cm⁻¹, there was a vibration absorption peak of an aromatic hydrocarbon C=C skeleton, and the law was Y₂S₁ > F > Y₁ > Y₂S₂ > M > M₁; thus, the content was the highest in Longkou oil shale Y₂S₁, followed by Fushun oil shale, and the control coal sample in succession. At 3450 cm⁻¹, there existed intermolecular hydrogen bonding of a phenolic hydroxyl group, alcoholic hydroxyl group, or amino, and the law was M > F > M₁ > Y₁ > Y₂S₁ > Y₂S₂. In the 1800–1300 cm⁻¹ region, there were absorption peaks of the oxygen-containing group C=O, C=C skeleton in a benzene ring structure, and symmetric vibration of –CH₃, and the overall law was that the content of aliphatic hydrocarbons in the control coal samples was higher than those in Fushun oil shale and Longkou oil shales. To sum up, the six different samples mainly contained aliphatic hydrocarbons, aromatic hydrocarbons, oxygen-containing functional groups, etc. Aliphatic hydrocarbons and aromatic hydrocarbons belonged to hydrophobic groups, which repressed the wettability of oil shale dust, and this was the fundamental cause for surface hydrophobicity, but oxygen-containing functional groups like hydroxyl, carboxyl, and carbonyl endowed coal dust with certain hydrophilia.

Influence of Minerals on the Wettability of Oil Shale Dust and Coal Dust. As shown in Table 3 and Figure 4, XRD

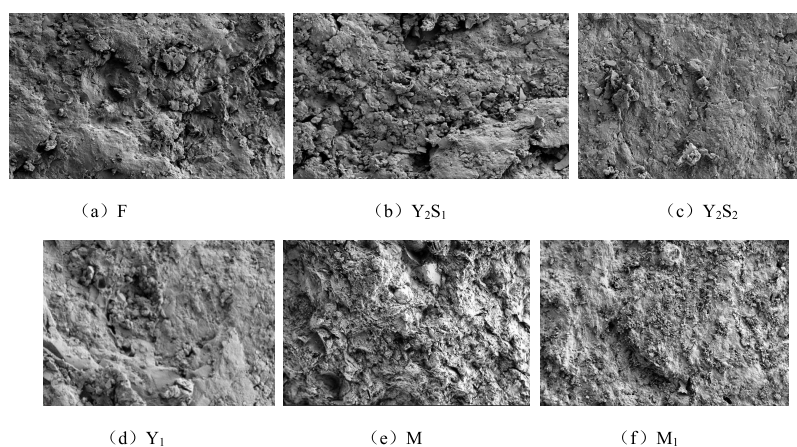


Figure 5. SEM graphs of different samples (8000 \times).

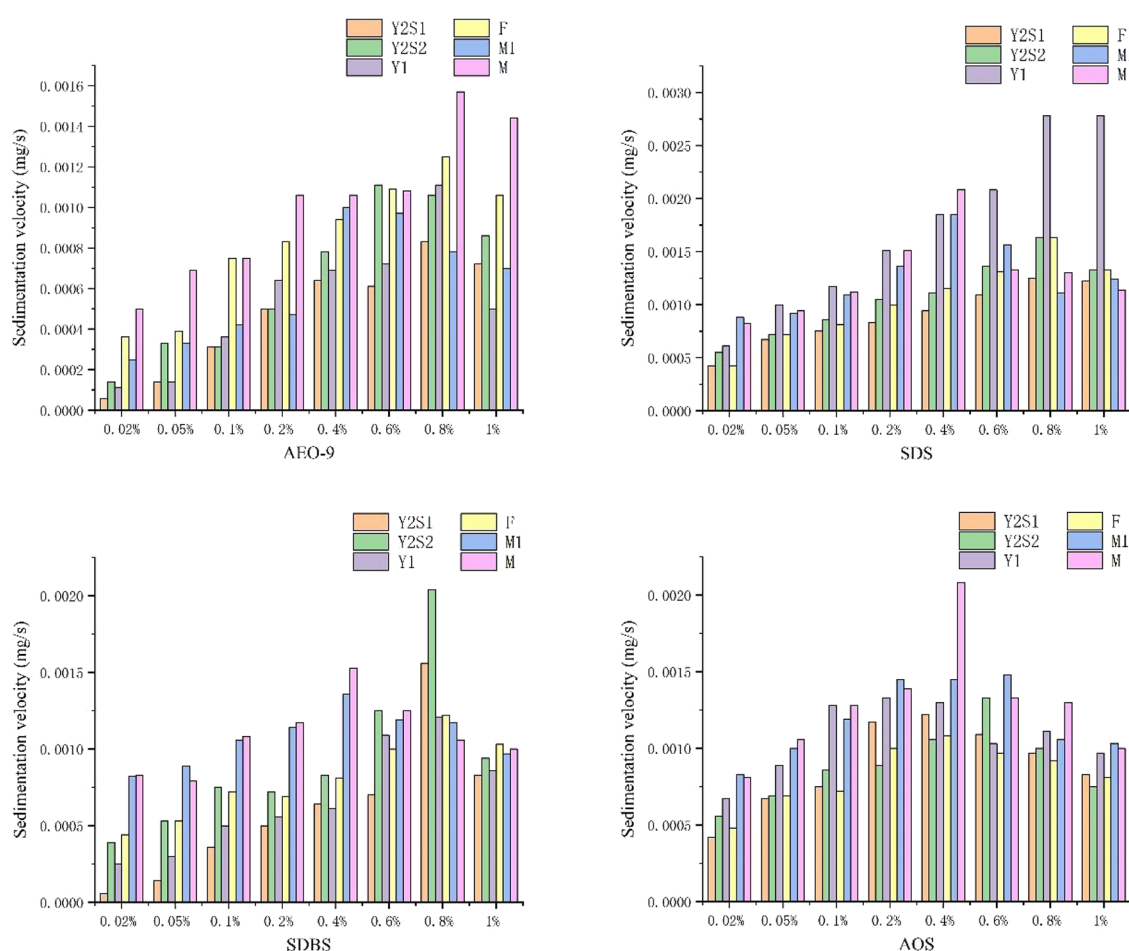


Figure 6. Sedimentation velocity of oil shale dust and coal dust with different surfactants.

results showed that Longkou oil shales had great differences from Fushun oil shale and coal samples in the content of minerals, where their SiO_2 contents were remarkably higher than those in Fushun oil shale F and coal samples M_1 and M. Meanwhile, the oil content in Longkou oil shale Y_2S_2 was the highest with the SiO_2 content also reaching the highest value (91.3%), while the lowest content was 75.2%. Longkou oil shale dust has high content of SiO_2 , and free SiO_2 in the dust has a great impact on human health and will cause miners to suffer from severe silicosis. Therefore, there is an urgent need to solve the problem of oil shale dust suppression.

Influence of the Surface Structure on the Wettability of Oil Shale Dust and Coal Dust. Wetting of oil shale dust is a complicated process involving the interaction among solid, liquid, and gas (three phases).²⁹ Oil shale dusts with different oil contents have different porous structures, which may influence the wetting ability of the reagent to oil shale.

Scanning electron microscopy (SEM) was performed to observe the surface micromorphology of oil shale dust and coal dust. Compared with coal, the pores of oil shales were mainly semiclosed pores, which had poor connectivity, poor separation, a small pore size, a large specific surface area, and a rougher

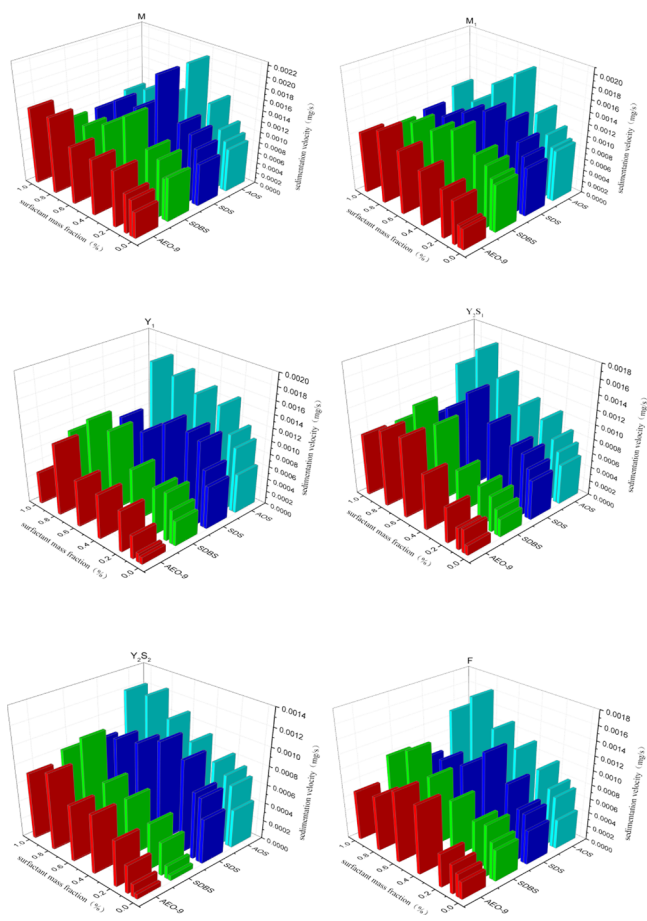


Figure 7. Sedimentation velocities of the same sample in different surfactants.

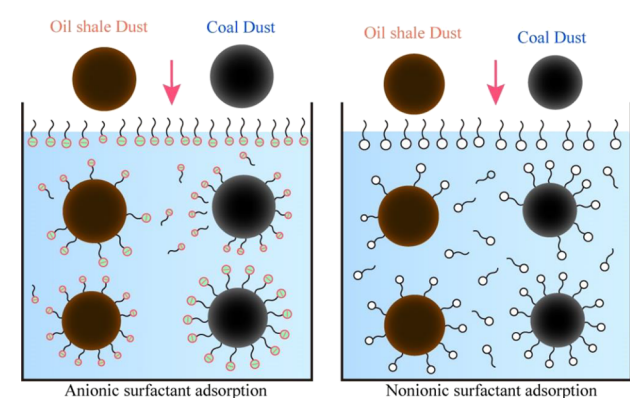


Figure 8. Schematic chart of adsorption of anionic surfactant and nonionic surfactant solutions on the surface of oil shale dust compared with coal dust.

surface, making the wettability worse. The surface image of Fushun oil shale was similar to those of Longkou oil shales, all having rough surfaces. As the oil content increased, the surface roughness of Longkou oil shale dust was reduced. The rough dust surface made it more difficult for solution diffusion on the dust surface and weakened the wetting ability of liquid for dust (Figure 5).

Influence of Surfactants on Sedimentation Velocities of Oil Shale Dust and Coal Dust. The experimental results of sedimentation velocity are shown in Figure 6. After the surfactant was added, the wetting effects of oil shale dust and

coal dust were both improved, and as the surfactant concentration increased, the sedimentation velocities of oil shale dust and coal dust were gradually increased. Generally speaking, coal samples reached the optimal sedimentation velocity under 0.4% concentration of the surfactant, but oil shales could realize this only under 0.6–0.8% concentration. The same surfactant had different wetting abilities for oil shales with different oil contents and coal dust. First, the sedimentation velocity of coal dust in the surfactant was higher than that of oil shale dust, mainly because there were more oxygen-containing functional groups in coal dust and fewer polycyclic aromatic hydrocarbons and aliphatic hydrocarbon in their structures than those of oil shale dusts. Second, the sedimentation velocity of Longkou oil shale dust was slowed down as the oil content increased, the sedimentation velocity of Longkou oil shale Y_2S_2 was the lowest, and the rule was $Y_1 > Y_2S_1 > Y_2S_2$. This was because with the change in oil content, the number of rings of aromatic hydrocarbons was gradually increasing, while that of oxygen-containing functional groups was declining, thus leading to the change in surface hydrophobicity of oil shale.

Figure 7 presents the sedimentation velocities of oil shale dust and coal dust in different surfactants. On the whole, anionic surfactants had stronger wetting ability to both oil shale dust and coal dust than nonionic surfactants, where the wettability law of oil shale dust and brown coal by several surfactants was $AOS > SDS > SDBS > AEO-9$, and the wettability law of bituminous coal dust was $SDS = AOS > SDBS > AEO-9$.

Figure 8 shows the schematic chart of adsorption of anionic surfactant and nonionic surfactant solutions on the surface of oil shale dust compared with coal dust. In the adsorption process, the hydrophobic surface of the oil shale dust and coal dust interacted strongly with the hydrophobic groups of the surfactant. Therefore, the tail hydrophobic group of the surfactant orientated to the dust surface and the head hydrophilic group to the solution. Since the hydrophilic group of the surfactant extended into the solution after adsorption, the hydrophilicity of oil shale dust and coal dust was enhanced, and the wetting performance of the solution to oil shale dust and coal dust was significantly improved. In addition, the wettability of oil shale dust was weaker than that of coal dust.

CONCLUSIONS

- (1) Compared with coal, oil shale has lower contents of water, volatile matter, and fixed carbon, while the oil content and ash content are higher. The wettability of oil shale is positively correlated with water and fixed carbon contents and negatively correlated with ash content. The volatile content has little effect on the wettability of the samples. Through scanning electron microscopy observation of the surface structure of oil shale and coal samples, it can also be found that the surface structure of oil shale is mainly composed of semiclosed pores and is a rough surface with poor connectivity and poor wettability, and it is more difficult for the solution to wet the surface of oil shale.
- (2) Through FTIR results, we can see that organic functional groups are also the important factors affecting the wettability of oil shale. Oil shale contains more aliphatic and aromatic hydrocarbons and less oxygen-containing functional groups, which lead to the decrease in hydrophilicity and worse wettability than coal dust. According to the XRD results, the SiO_2 content of Longkou oil shale is higher than that of coal dust, and the

SiO₂ content of Y₂S₂ with the highest oil content is 30.43% higher than that of M₁ and 50.91% higher than that of M. The high SiO₂ content makes oil shale dust more harmful to human health than coal dust.

- (3) It is found through experiments that the solution after adding surfactants can effectively wet oil shale, but the sedimentation rate of oil shale dust is lower than that of coal dust. The sedimentation effect of adding anionic surfactants is better than nonionic surfactants, and for oil shale dust, AOS has the best effect.

MATERIALS AND METHOD

Experimental Materials. Oil shales were collected from Longkou in Shandong and Fushun in Liaoning (F). Samples

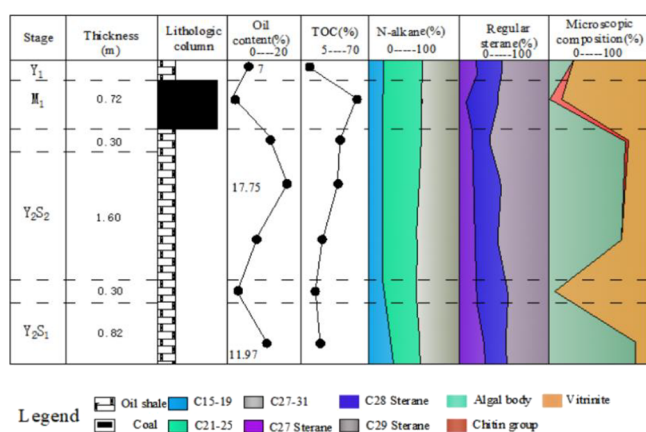


Figure 9. Vertical evolution graph of Longkou oil shale and coal organic matter.

from Longkou were divided into brown coals M₁, Y₁, Y₂S₂, and Y₂S₁ according to their buried depth and oil content, where their oil contents were sorted as Y₂S₂ > Y₂S₁ > Y₁ > M₁ (Figure 9). In addition, bituminous coal (M) from Jining in Shandong was taken as the control sample. The six samples were crushed and sieved into dusts with granularity being smaller than 200 meshes (0.074 mm).

Due to the special exploitation environment, surfactants should be nontoxic, nonirritant, nonflammable, and dissolvable in water with low cost.³⁰ A study shows that anionic surfactants and nonionic surfactants have strong wetting abilities for coal dust.³¹ To understand the influence of surfactants on the wettability of oil shale dust, three anionic surfactants and one nonionic surfactant were selected in this study as seen in Table 4.

Experimental Method and Facilities. The proximate analysis of oil shales and coal samples was carried out using a WS-G818 fully automatic proximate analyzer manufactured by Willson Technology Co. Ltd. in Changsha, in accordance with the China coal industry standard MT/T 1087-2008 *Methods for Proximate Analysis of Coal by Instruments*.³²

Table 4. Surfactants Used in the Experiment

reagent name	abbreviation	molecular formula	grade	category
sodium dodecyl benzene sulfonate	SDBS	C ₁₆ H ₂₉ NaO ₃ S	analytical reagent	anionic
sodium dodecyl sulfate	SDS	C ₁₂ H ₂₅ SO ₄ Na	analytical reagent	anionic
sodium alpha-olefin sulfonate	AOS	RCH=CH(CH ₂) _n SO ₃ Na, R = C14–16	analytical reagent	anionic
primary alcohol ethoxylate	AEO-9	RO(CH ₂ CH ₂ O) ₉ H, R-12	analytical reagent	nonionic

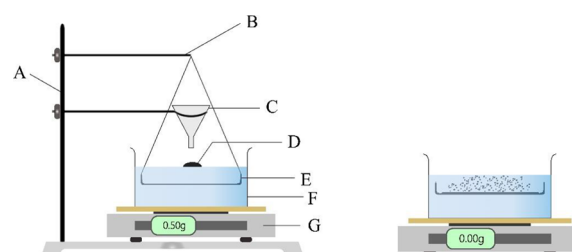


Figure 10. Schematic diagram of the sedimentation experiment, including an iron support (A), locating ring (B), glass funnel (C), coal sample (D), glass container (E), weighing bottle (F), and electronic balance (G).

The contact angle experiment is one of the main methods to determine the wettability of dust. However, due to the special structure of oil shale, the surface of oil shale absorbs water and cracks during the contact angle experiment, so the contact angle cannot be formed. Therefore, we chose the sedimentation experiment to test the wettability of oil shale.

The traditional sedimentation experiment was optimized according to the standard of *Determination Method of Dust-Sedimentation Performance for Mine* (MT 506-1996).³³ The change in sedimentation mass was recorded with an electronic balance as seen in Figure 10. The electronic scale was used to weigh 500 mg of samples; different reagents of different concentrations were prepared according to the experimental requirements: second as the time unit and milligram as the mass unit. The sedimentation mass and time of samples (namely, the mass of samples falling into the vessel bottom) to be completely immersed were recorded after the dust samples contacted the solution until the whole dust sample was immersed into the solution or the sedimentation mass shown in the balance no longer changed. Every experiment was carried out three times to take the average value. The deviation between measured values and the average value should be less than 7%.⁵

The infrared spectroscopic analysis was implemented using a Nicolet iS50 Fourier transform infrared spectrometer produced by Thermo Fisher Scientific Co. Ltd. in the USA. The infrared spectra of dust samples were determined through the potassium bromide pellet technique. The sample and KBr (chromatographically pure) were dried in a 105 °C drying oven for 2 h. Then, the sample and KBr were taken out and placed into a dryer for cooling to room temperature. Two milligrams of the sample and 200 mg of KBr were ground into the size smaller than 2 μm using an agate mortar by the proportion of 1:100. A proper amount of mixed powder was placed into an abrasive tool and pressed into a transparent wafer. The wafer was placed into the sample chamber of the spectrometer for testing.³⁴

Oil shales were tested via a Rigaku Ultima IV X-ray diffractometer produced by Japanese Rigaku Cooperation. It is required that granularity (about 45 μm) should be uniform and the usage of samples should not be less than 0.5 g; powder samples were fixed using a backpressure method for testing.⁵ The measuring planes of the produced samples should be tight,

flat, and smooth with small orientation. The sample should be immediately determined to mitigate the influence of water-absorbing quality.

Surface morphologies of oil shale samples and coal samples were tested by using an Apreo scanning electron microscope produced by FEI Co. Ltd. in the USA.

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Notes

The authors declare no competing financial interest.

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