Micromechanism Analysis of Surfactant Wetting of Coal Based on $^{13}$C NMR Experiments

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ABSTRACT: With the gradual improvement in coal mine mechanization and automation, the dust concentration at production sites is increasing significantly as the production efficiency improves, which not only poses a substantial threat to the occupational safety and health of workers but also affects the safe production and social stability of mines. At present, wet dust removal is the most economical and effective technical dust removal measure. However, most coal seams in China have poor wettability, unclear microscopic wetting mechanisms, and poor dust removal effects. Therefore, based on experiments and numerical analysis, this paper qualitatively studies the influencing factors of surfactants on coal wettability and quantitatively constructs an innovative evaluation model of the influence of the microstructure of coal and surfactants on wettability. First, based on $^{13}$C NMR experiments, the structural parameters of coal and several surfactants were obtained. Second, the wettability relationship between the coal and the surfactants was determined by optical titration, and the key factors affecting the wettability of coal dust and the wettability of the surfactants were selected. Then, using numerical analysis and function fitting analysis and combining the structural parameters with the coal wetting results, the relationship between the microstructure of the surfactants on different kinds of coal and the wettability of the coal samples was established. The results show that the ether group, the phenol or aromatic ether carbon, the fatty methyl group, and the aromatic methyl group in the surfactants have a substantial influence on the wettability. The research results can provide scientific guidance for the development of efficient and environmentally friendly compound dust suppressants to realize clean production in mines.

1. INTRODUCTION

In recent years, the mining depth of coal has been continuously increasing, and the mining level of mechanization and automation has been gradually improving. The subsequent natural disasters in mine production have become increasingly prominent. Dust disasters in underground operation areas are especially severe and pose substantial threats to the safety of mine production and the physical and mental health of coal miners.1,2 According to statistics, in the working area of fully mechanized mines, if there are no dust control measures, the instantaneous original total dust concentration of individual working sites can be as high as 4000−6000 mg/m$^3$ when the shearer is working or when the shearer is working together with frame removal and other processes. A high degree of mechanization, fast footage, a large amount of dust per unit time, and a high concentration of dust migration and dispersion under the action of air currents can lead to serious dust pollution in comprehensive excavation sites.3,4 Even if dust control measures are adopted, the operating environment of most working surfaces is still not ideal,5−8 and dust concentrations often exceed the national upper limit. Due to the complex structure of coal, its wettability can involve many factors, including the contents of fixed carbon, water, and ash, the internal pore structure, the microscopic carbon structure, and the functional groups on the coal surface.9 In addition, the wettability performance of coal is closely related to the properties of selected coal wetting liquids. The study of coal dust wetting has not been formalized into a system due to the complexity of the wetting mechanism, so breakthroughs in dust removal work are difficult. In terms of the coupling effect of the wetting liquid and coal dust, it is necessary to determine the microstructure parameters of surfactants and coal.11,12

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groups in coal and reached the conclusion that the primary factor affecting the wettability of coal is the carboxyl group. D. V. Keller, F. Osasere Orumwense, and others determined the wettability of coal and the carbon in coal, the acid–base of the wetted liquid, and the influence of minerals in coal on wettability based on a contact experiment with coal dust and water. Ofori et al. used a relatively novel test method to study coal wettability; they used image analysis technology, petrography, and contact angle measurement experiments to comprehensively evaluate the wettability of coal and found that the hydrophobicity of each component of coal was closely related to the wettability.

Many domestic scholars have made substantial progress in research on the mechanism of coal wettability. Domestic scholars Yang Linjiang and Ouyang Yunli et al. proposed that the wettability of coal is affected by various characteristics and studied the effect of many factors, such as the coal rank, oxygen-containing functional groups, surface electricity, and the composition of coal, on the wettability of coal. In the process of coal seam water injection, Jin Longzhe and Wang Qingsong et al. explored the movement dynamics and process of water in a coal seam, analyzed the wetting process of a coal body surface based on the theory of interface chemistry, and obtained the conditions for a coal body to be wetted by itself. Yang Jing, Tan Yunzhen et al. studied the wetting mechanism of coal dust by means of infrared spectrum tests, electrophoresis tests, and forward osmosis tests and found that the surface tension of the wetting liquid is not the only factor affecting the wettability of coal dust; the magnitude of the solid–liquid interfacial tension and the hydrophobicity, electricity, structure, and properties of the surfactant also affect this parameter. Li Qingzhao et al. explored the influence of particle size and the fractal dimension of the sample surface structure on coal dust wettability. Their results showed that as the fractal dimension of the particle size increased, the particle size in the middle of the particle decreased, and the wettability contact angle on the surface increased. Further, their results showed that this conclusion was significant for coal dust with particle sizes smaller than 10 μm. Zhou and Xu et al. used contact angle and NMR experiments to explore the influence of the carbon structure in coal on coal wettability and fitted the contribution of the carbon structure in coal to its wettability.

Regarding the influencing factors of the wetting ability of surfactants on coal dust, James O et al. studied various factors affecting the wetting rate of coal dust by measuring the wetting rate of the wall. The experimental results showed that the wetting rate is mainly related to the temperature, the particle size, the composition of the coal dust, and the types and characteristics of the surfactants used. Howard W et al. found that the wettability of coal is greatly related to the type and content of minerals contained in the coal. In a wettability experiment on coal, Howard W added sodium salt or potassium salt to the anionic surfactant. The results of the experiment showed that the affinity of the surfactant and coal wettability are greatly enhanced, and the wettability of coal is significantly improved. Bimal P. Singh tested the adsorption kinetics and adsorption isotherm of a surfactant acting on the surface of coal dust. The experimental results showed that the adsorption kinetics and adsorption isotherms were related to the Langmuir model. In addition, their results revealed the interaction between coal and surfactants at the microworld and the reason for the changing wettability. Sun Yinyu, Nie Rongchun et al. analyzed raw coal by means of contact angle experiments, infrared spectrum experiments, and surface tension experiments. The main factors affecting the wettability of coal included not only the coal itself but also the type, concentration, surface tension, and surface structure of the surfactant water solution. Xu Haihong et al. explored the influence of different kinds of surfactants on the wettability of coal through coal flotation experiments. Their results showed that the internal structure of a surfactant has an important influence on its ability to wet coal. Zhao Di compounded a variety of surfactants according to the analysis of basic physical and chemical properties of a variety of experimental coal samples. Based on molecular thermodynamics and surface chemistry, Ma Yanling et al. analyzed the principle of the surface modification of coal by surfactants from a microperspective and explained that the addition of surfactants reduced the surface tension of water and the interfacial tension between water and coal, increased the molecular force between water and coal, reduced the free energy of the system, and increased the wettability of coal.

Coal mine dust not only seriously threatens the occupational safety and health of underground workers but also affects the safety and social stability of mines. To realize the rapid, healthy, and sustainable economic development of coal enterprises and to maintain the occupational safety, health, and stability of employees, it is necessary to control the dust in the mining face. The microphysical and chemical structures of coal are an important factor affecting the wettability of coal. Therefore, to solve the problem of poor chemical dust suppression effects in the field of industry and mining, it is not enough to study only coal; the surfactants must also be studied. It can be said that the use of a chemical dust suppression agent does significantly improve the underground working environment. However, we cannot ignore the human and material resources involved with the proportion of dust suppression agents, which is a serious waste of financial resources. At the same time, due to the particularity of fully mechanized mining faces, there are some technical difficulties in dust control. Therefore, the micro-mechanism analysis of surfactant wetting coal can provide theoretical support for the application of existing dust control technology systems in mining faces and effectively reduce the dust concentration in the working face, create a suitable working environment for workers, and ensure safe, clean, and efficient production in mines.

2. MATERIALS AND METHODS

2.1. Selection of Materials. To ensure that the coal samples were representative and to ensure their richness, coal samples with different coal grades representing six regions in China, including Beizao lignite, Huangtao nonsticky coal, Jinrun gas coal, Xinjulong fat coal, Xinzhi coking coal, and Yangquan anthracite, were selected (Table 1).

Based on a study of many mines at home and abroad, nine kinds of surfactants were ultimately selected by comprehensively considering the cost, use effect, environmental protection, and various other factors, as shown in Table 2.

2.2. Nuclear Magnetic Resonance Experiments. In this study, a BRUKER AVANCE III 400 nuclear magnetic resonance (NMR) apparatus was used to carry out experiments. The experimental equipment is shown in Figure 1. In the experiment, cross-polarization magic-angle spinning (CPMAS) rotation technology and total rotation of spinning
a 300 mL was crushed, passed through a 200-mesh sieve, and placed in the coal samples were deashed. First, 30 g of a coal sample mL of deionized water, 50 mL of hydrochloric acid (37% by Table 3. Coal Sample Test Conditions

| number | test parameters | condition |
|--------|-----------------|-----------|
| 1      | magic-angle speed | 5 kHz     |
| 2      | resonant frequency | 100.38 MHz |
| 3      | sampling time | 0.0026 s |
| 4      | spectral width | 39682 Hz |
| 5      | spectral width | 2 s |
| 6      | number of scans | 10240–20480 times |
| 7      | cross-polarization contact time | 3000 μs |

sidebands (TOSS) technology were used to test the coal samples. The test conditions are shown in Table 3. Due to the strict requirements of $^{13}$C NMR experiments, the coal samples were deashed. First, 30 g of a coal sample was crushed, passed through a 200-mesh sieve, and placed in a 300 mL flat-bottom flask. In another 300 mL beaker, 100 mL of deionized water, 50 mL of hydrochloric acid (37% by mass), and 50 mL of hydrofluoric acid (37% by mass) were sequentially added to prepare a mixed acid solution. The prepared mixed acid was slowly poured into the flask several times and shaken repeatedly to ensure that the inner wall of the flask was free of coal sample residues above the liquid level, the flask was sealed with tin paper, and the water bath was heated to 50 °C and held for 12 h. Then, the solution was filtered, the coal sample was washed with deionized water until the pH value of the filtrate was 7, and the filtered coal sample was put into a vacuum drying oven and dried at 60 °C for 12 h to obtain an experimental sample after ash removal. The selected surfactants were all analytically pure or chemically pure and water soluble. Therefore, the experimental pretreatment of the surfactant samples during NMR was relatively simple. Approximately 10 μL of a sample were dissolved in 0.5 mL of heavy water, and the mixture was allowed to stand for 12 h. After the sample was completely dissolved in heavy water, the experiment was started.

2.3. Wettability Test of the Coal and Surfactants. In this paper, a DSA100-type optical droplet morphology analysis system was used to measure the dynamic contact angle of the surfactants and coal, as shown in Figure 2. The operation steps were as follows: 2.3 g of pulverized coal with a particle size of approximately 2 μm was pressed into a cylindrical sample with a diameter of 13 mm and a thickness of 1 mm using a tablet press under a pressure of 20 MPa. The sample to be tested was fixed on the test platform, and the syringe was installed. The position of the needle and the shape of the droplet were controlled by the control panel, and the contact angle was measured after the baseline had been measured. The final result was then displayed by the instrument.

3. RESULTS AND ANALYSIS

3.1. $^{13}$C NMR Spectra and Carbon Structural Analysis of the Coal. 3.1.1. Analysis of the $^{13}$C NMR Spectra of the Coal. The spectrogram obtained with $^{13}$C NMR experiments can provide the structural parameters of coal, which are mainly various aromatic carbon structures and aliphatic carbon structures. Therefore, to obtain the relative content of carbon with different structures, the superposed peaks in the obtained spectrogram must be subjected to a reasonable peak-splitting treatment. Currently, computers are widely used to fit spectrograms and to destack peaks. In this study, the $^{13}$C NMR spectrum of the sample was fitted using the NUTS98 software; a Gaussian function was used for the smoother peaks, and a Lorentzian function was used for the sharp peaks. Figures 3–8 were obtained after peak-splitting fitting of the $^{13}$C NMR spectra of the six kinds of coal samples. It can be seen from the graphs that the fitting spectra indicated by red lines and the experimental spectra are very well matched, which shows that the peak-splitting fitting done in this paper is reasonable.

As seen from the above figures, with increasing coal metamorphism, the NMR spectra also change regularly, mainly showing that the higher the rank of coal is, the greater the proportion of the aromatic carbon is, and the lower the proportion of aliphatic carbon. After peak splitting occurs, the aromatic carbon peak group is sharp and highly convex, and its top is composed of multiple peaks that are formed by the overlapping absorption peaks of protonated aromatic carbon and bridged aromatic carbon, with a close chemical shift.
Compared with the peaks indicating an aromatic carbon structure, the lipid carbon peaks are mostly low and scattered peaks, which shows that all kinds of lipid carbon structures are distributed in the carbon structure of coal, but the content is relatively low. The specific structural parameters are shown in Table 4.

In the carbon structure of coal, the content of aromatic carbon occupies an absolute advantage; the content in lignite reaches 64.36%, and it shows an increasing trend with increasing coal metamorphism, and the content in anthracite can be greater than 90%. Additionally, during the evolution process, the protonated carbon content increases, while the content of aromatic ring heteroatoms decreases. In contrast, during the evolution process, the proportion of aliphatic carbon in the carbon structure of coal decreases continuously, and each structural parameter basically conforms to the law of carbon enrichment and deoxidation during coal metamorphism.

3.1.2. Analysis of the Aromatic Carbon Structure of the Coal. According to the structural parameters of the aromatic carbon of the six coals with different metamorphic degrees, the aromatic ring carbon, nonprotonated carbon, protonated...
carbon, phenol or aromatic ether carbon, alkyl-substituted aromatic carbon, and bridged aromatic carbon are classified, as shown in Figure 9.

The aromatic ring carbon content is one of the parameters that can quantitatively reflect the organic structure of a sample and is an important component of the aromatic carbon structure. Additionally, coal is directly related to the evolution degree of coal. As shown in Figure 9a, with increasing coal metamorphism, an obvious increasing trend of aromatic carbon can be seen. The growth rate of aromatic carbon is relatively slow from lignite metamorphism to fat coal, while the growth rate of aromatic carbon is relatively obvious from fat coal metamorphism to anthracite. Therefore, it can be inferred that for coals with different metamorphic degrees, the increase in the aromatic structure does not conform to a linear relationship, and its growth rate is a gradual and accelerated process.

Figure 7. Curve-fitted $^{13}$C NMR spectra of JM.

Figure 8. Curve-fitted $^{13}$C NMR spectra of WY.

Figure 9. Structural parameters of the aromatic carbon in the samples: (a) $f'$ and $f''$ variation maps of 6 coals with different metamorphic degrees, (b) $f'$ and $f''$ variation maps of 6 coals with different metamorphic degrees, and (c) $f'$, $f'$, $f''$, and variation maps of 6 coals with different metamorphic degrees.

### Table 4. Structural Parameters of the Coals$^a$

| coal samples | $f'_{\text{C}}$ | $f''_{\text{C}}$ | $f'_{\text{H}}$ | $f''_{\text{H}}$ | $f'_{\text{O}}$ | $f''_{\text{O}}$ | $f'_{\text{P}}$ | $f''_{\text{P}}$ | $f'_{\text{B}}$ | $f''_{\text{B}}$ |
|--------------|----------------|----------------|----------------|----------------|----------------|----------------|----------------|----------------|----------------|----------------|
| HM           | 64.36          | 4.3            | 60.06          | 16.99          | 43.07          | 5.97           | 0              | 11.02          | 38.92          | 5.4            | 27.29          | 6.23            |
| BN           | 65.68          | 3.1            | 62.58          | 31.02          | 31.56          | 4.01           | 16.9           | 10.11          | 37.41          | 3.82           | 31.73          | 1.86            |
| QM           | 66.56          | 2.5            | 64.06          | 23.27          | 40.79          | 4.94           | 6.07           | 12.26          | 35.95          | 8.03           | 24.55          | 3.37            |
| FM           | 70.2           | 1.9            | 68.3           | 24.22          | 44.08          | 6.12           | 9.05           | 9.05           | 31.69          | 5.2            | 26.2           | 0.29            |
| JM           | 79.65          | 4.62           | 75.03          | 22.66          | 52.37          | 4.22           | 7.2            | 11.24          | 24.98          | 1.14           | 23.84          | 0               |
| WY           | 90.54          | 2.32           | 88.22          | 38.64          | 49.58          | 2.7            | 9.12           | 26.82          | 11.77          | 0.0            | 10.63          | 1.14            |

$^a$Note: $f'_{\text{C}}$, aromatic carbon; $f''_{\text{C}}$, carbonyl carbon with chemical shift > 165 ppm; $f'_{\text{H}}$, aromatic ring carbon; $f''_{\text{H}}$, nonprotonated carbon; $f'_{\text{O}}$, protonated carbon; $f'_{\text{P}}$, phenol or aryl ether carbon; $f''_{\text{P}}$, alkyl-substituted aromatic carbon; $f'_{\text{B}}$, bridged aromatic carbon; $f'_{\text{H}}$, fatty carbon; $f''_{\text{H}}$, fatty (aromatic) methyl; $f'_{\text{B}}$, quaternary carbon or methylene; $f''_{\text{B}}$, oxygen bonded carbon.
Protonated aromatic carbon and nonprotonated aromatic carbon reflect the ratio of hydrogen atoms to carbon and the content of heteroatom carbon in aromatics, respectively. Figure 9b demonstrates that protonated carbon also shows an increasing trend as the coal deterioration degree increases. However, nonprotonated aromatic carbon does not show regularity with increasing coal metamorphism.

As can be clearly seen in Figure 9c, the content of bridged aromatic carbon increases with increasing coal metamorphism, indicating that the previously smaller condensed aromatic ring forms a condensed aromatic ring unit with a greater extension through the connection of new bridgehead carbon. Additionally, the change trend of bridging aromatic carbon is relatively stable, and with increasing coal metamorphism, there is a relatively stable growth trend.

3.1.3. Structural Analysis of the Lipid and Carbon Contents in the Coal. The fatty carbon structures of the six coals with different metamorphic degrees are shown in Figure 10.

![Figure 10](image_url)

**Figure 10.** Structural parameters of the aliphatic carbon in the samples.

The changes in the fatty carbon structural parameters of the six coals with different metamorphic degrees are shown in the graph in Figure 10. Among them, the content of quaternary carbon or methylene decreases with increasing coal metamorphism. However, the oxygen-bonded carbon with a lower content also shows a downward trend with the change in coal metamorphism. However, the lipid (aromatic) methyl group does not show a relatively obvious change trend.

Although the existing forms of coals with different metamorphic degrees are basically the same, the differences in the carbon structure content lead to great differences in structure.\(^3\) According to the above analysis, the continuous increase in aromatic ring carbon can represent the improvement of coal aromatization. The increase in the protonated carbon also makes the aromatic carbon increasingly concentrated in the protonated carbon, and the functional groups on the aromatic ring are increasingly less concentrated. The increase in the content of bridged aromatic carbon also explains the change in the aromatic structure; that is, a thick condensed aromatic ring is formed by joining a thick aromatic ring. Additionally, with decreasing seasonal carbon or methylene and oxygen lipolycarbon, the long alkyl side chain breaks into a short chain, the lipolycarbon chain continues to fall off, and the number decreases.

3.2. \(^{13}\)C NMR Spectra and Carbon Structure Analysis of the Surfactants. 3.2.1. \(^{13}\)C NMR Spectral Analysis of the Surfactants. The surface activity and purity of the polymer are relatively simple, and in the obtained NMR spectra, the peaks obtained by each carbon structure are rod-shaped. Therefore, the surfactant atlas processing method was relatively simple. The obtained atlas was imported into the nuclear magnetic analysis software MestRe-C, each peak in the atlas was integrated, and a peak was selected as a reference to further obtain the relative area of each peak. After the relative area of each peak was obtained, statistics and records were created according to the carbon structure attribution. The carbon structure parameters of the surfactants are shown in Table 5.

Although there is no logical evolution between the surfactants, it can be seen from the above table that the content of aliphatic carbon accounts for the vast majority of the carbon structure of the surfactants, and some surfactants do not have aromatic carbon structures.

3.2.2. Analysis of the Aromatic Carbon Structure of the Surfactants. The structural parameters of the aromatic carbon of the nine surfactants are classified and shown in Figure 11.

According to the aromatic carbon structure of the surfactants, it can be concluded that the aromatic carbon content of a given surfactant is small, and the Q6 and Q8 surfactants do not contain aromatic carbon structures. The Q1, Q2, Q7, and Q9 surfactants do not contain carbonyl carbon structures, and the Q3 and Q4 surfactants do not contain aromatic ring structures. It should be noted that the aromatic carbon structure content of the Q9 surfactant is the highest among the selected surfactants, and all of them are aromatic ring parts, in which the protonated carbon content is 4.48, the nonprotonated carbon content is 29.06, and the nonprotonated carbon bridges aromatic carbon. This shows that the aromatization degree of the Q9 surfactant is higher and more stable.

**Table 5. Carbon Structure Parameters of Surfactants**

| Sample | \(f_a\) | \(f_e\) | \(f_b\) | \(f_d\) | \(f_c\) | \(f_{al}\) | \(f_{am}\) | \(f_{aq}\) | \(f_{al}\) | \(f_{am}\) | \(f_{aq}\) |
|-------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| Q1    | 6.90   | 6.90   | 1.51   | 5.39   | 1.51   | 93.10  | 4.76   | 19.98  | 68.36  |        |        |
| Q2    | 7.77   | 7.77   | 1.69   | 6.08   | 0.96   | 0.73   | 92.23  | 3.62   | 9.95   | 78.66  |        |
| Q3    | 8.93   | 8.93   | 1.69   | 6.08   | 0.96   | 0.73   | 91.07  | 4.43   | 61.22  | 25.42  |        |
| Q4    | 2.27   | 2.27   | 1.32   | 1.32   | 0.44   | 0.87   | 97.73  | 3.42   | 31.49  | 62.82  |        |
| Q5    | 2.85   | 1.53   | 1.32   | 1.32   | 0.44   | 0.87   | 97.15  | 3.42   | 31.49  | 62.82  |        |
| Q6    | 10.81  | 10.81  | 10.81  | 10.81  | 10.81  | 10.81  | 10.81  | 10.81  | 10.81  | 10.81  |        |
| Q7    | 10.81  | 10.81  | 10.81  | 10.81  | 10.81  | 10.81  | 10.81  | 10.81  | 10.81  | 10.81  |        |
| Q8    | 0.00   | 0.00   | 0.00   | 0.00   | 0.00   | 0.00   | 0.00   | 0.00   | 0.00   | 0.00   |        |
| Q9    | 33.54  | 33.54  | 29.06  | 4.48   | 29.06  | 66.46  | 5.27   | 41.62  | 19.57  |        |        |

[3] According to the aromatic carbon structure of the surfactants, it can be concluded that the aromatic carbon content of a given surfactant is small, and the Q6 and Q8 surfactants do not contain aromatic carbon structures. The Q1, Q2, Q7, and Q9 surfactants do not contain carbonyl carbon structures, and the Q3 and Q4 surfactants do not contain aromatic ring structures. It should be noted that the aromatic carbon structure content of the Q9 surfactant is the highest among the selected surfactants, and all of them are aromatic ring parts, in which the protonated carbon content is 4.48, the nonprotonated carbon content is 29.06, and the nonprotonated carbon bridges aromatic carbon. This shows that the aromatization degree of the Q9 surfactant is higher and more stable.

ACS Omega 2021, 6, 1378–1390
3.2.3. Analysis of the Lipid Carbon Structure of the Surfactants. The structural parameters of the fatty carbon of the nine surfactants are classified and shown in Figure 12. The proportion of the aromatic carbon structure in the surfactants is relatively small, while the proportion of aliphatic carbon is relatively rich. Here, \( f_a \) represents the relative content of the aliphatic methyl group in coal, \( f_{nc} \) represents the quaternary carbon or methylene group in coal, and the relative content of the methylene group, and \( f_{al} \) represents aliphatic carbon in contact with oxygen in coal. \( f_{al} \) accounts for a high proportion of the lipid carbon structure of the Q1 surfactant, which indicates that this surfactant contains a relatively rich branched-chain structure, and \( f_{nc} \) accounts for the majority of the carbon structure, indicating that this surfactant contains a large amount of oxygen elements. The structure of the Q2 and Q4 surfactants is relatively simple and similar to that of the Q1 surfactant, but compared with Q1 and Q2, the oxygen elements are richer, and the branched structure is relatively simple. However, the branched-chain structure of the Q4 surfactant is richer and more complex. The content of \( f_{al} \) in the Q3 surfactant accounts for the majority of the carbon structure, but the content of methyl is relatively small, which indicates that although this surfactant contains a rich branched-chain structure, the end of the branched chain is mostly connected with oxygen-bonded carbon. As shown in the above figure, the lipid carbon structure of the Q6 and Q9 surfactants is similar to that of the Q3 surfactant. The Q5 surfactant contains fewer oxygen elements, and the aliphatic carbon structure is mostly rich in branched-chain structures and has a higher content of methyl. The structure of the lipid carbon in the Q7 surfactant is quite special and is entirely oxygen-bonded lipid carbon. Therefore, it can be inferred that the structure of the Q7 surfactant is relatively simple. The Q8 surfactant has extremely rich branched structures.

3.3. Determination of the Wettability of the Coal and Surfactants. According to the literature, the micelle concentration is reached when the surfactant concentration reaches 0.06%. Therefore, the wettability of a surfactant solution with a concentration of 0.06% was measured in this experiment. To make the results more accurate, three groups of data were formed, and the average value was taken as the experimental result, as shown in Figures 13–18. From the above, it can be seen that the wettabilities of the various surfactants for different coals are quite different. For the water of the control experiment, the contact angle between the coal and the water gradually increases as the coal evolution degree increases before adding the surfactant, which
indicates that the affinity of coal to water gradually decreases as the coal evolution degree increases.34

For the Q1 surfactant, with increasing coal evolution, the affinity between the coal and the surfactant shows a trend of decreasing first and then increasing. For the Q2 surfactant, with increasing coal evolution, the affinity between the coal and the surfactant also increases. The wetting rule of the Q3 surfactant is similar to that of water, and the affinity decreases as the coal evolution degree increases. For the Q4 surfactant, it can be seen in the figure that the contact angle between lignite and anthracite is relatively high, and the affinity for nonstick coal, gas coal, and coking coal is similar, while the affinity for fat coal is strong. The Q5 surfactant shows a strong affinity for all of the coal samples. As the coal evolution degree increases, the affinity shows a downward trend, but it is not significant. The Q6 surfactant exhibits a wetting rule similar to that of the Q2 surfactant. As the coal evolution degree increases, the affinity shows an overall increasing trend. For the Q7 surfactant, as the coal evolution degree increases, the wettability shows a trend of decreasing first and then increasing. The wettability of the Q8 and Q9 surfactants is not very obvious. As the coal evolution degree increases, the affinity with coal first decreases, then increases, and then decreases.34–37

4. ANALYSIS OF THE MAIN INFLUENCING FACTORS OF SURFACTANT WETTING COAL DUST

4.1. Exploring the Main Factors Affecting Coal Dust Wettability from NMR. A correlation analysis was performed between the microscopic parameters of coal obtained by NMR and its wettability, and the analysis results are shown in Table 6.

From the analysis results, it can be seen that the carbon structure of coal obtained by NMR has a good correlation with the wettability of coal. Aromatic carbon, aromatic ring
Table 6. Correlation analysis between the Structural Parameters of Carbon and the Wettability of Coal

| contents                  | contact angle | aromatic carbon | carbonyl carbon | aromatic ring carbon | nonprotonated carbon |
|---------------------------|---------------|-----------------|-----------------|----------------------|----------------------|
| contact angle Pearson correlation | 1             | 0.941           | −0.865          | 0.940                | 0.156                |
| significance (bilateral)  | 0.005         | 0.026           | 0.005           | 0.768                |                      |

Table 7. Correlation Analysis between the Structural Parameters of the Carbon in the Surfactants and the Wettability for the Lignite Samples

| contents                  | protonated carbon | phenol or aryl ether carbon | alkyl-substituted aromatic carbon | bridging aromatic carbon | fatty carbon ratio |
|---------------------------|-------------------|-----------------------------|---------------------------------|-------------------------|-------------------|
| contact angle Pearson correlation | 0.904           | −0.812                      | −0.501                          | 0.922                   | −0.941            |
| significance (bilateral)  | 0.013            | 0.500                       | 0.311                           | 0.009                   | 0.005             |

| contents                  | fatty (aromatic) methyl | quaternary carbon or methylene | oxygen-bonded carbon |
|---------------------------|------------------------|---------------------------------|----------------------|
| contact angle Pearson correlation | −0.526          | −0.934                          | −0.947               |
| significance (bilateral)  | 0.283                 | 0.006                           | 0.004                |

Table 8. Fitting Result of the Wetting Effect of the Surfactant in the Lignite Samples

|                           | A               | B               | statistics          |
|---------------------------|-----------------|-----------------|---------------------|
|                           | value           | standard error  | value               | standard error      | reduced Chi-Sqr   | adj. R-square    |
|                           | 35.14248        | 2.14034         | −0.72355            | 0.18433             | 12.16357         | 0.6409           |

4.2. Analysis of the Main Factors Affecting the Wettability of the Surfactants from $^{13}$C NMR. Based on the correlation analysis method, the carbon structure parameters of the surfactant obtained by $^{13}$C NMR were correlated with the wettability of the surfactant. The specific analysis results are described below.

4.2.1. Analysis of the Main Factors of Surfactant Wettability in the Low Metamorphic Lignite Samples. The correlation analysis between the structural parameters of the carbon in the surfactants and the wettability for the lignite samples is shown in Table 7.

According to the analysis, the correlation between the carbon structure of the surfactant and the wettability of the lignite samples is not obvious, but the lipid (aromatic) methyl group shows a strong negative correlation with the wettability, with a significance coefficient of 0.004, which is less than 0.01.

In the wettability test of the lignite samples, the fatty (aromatic) methyl group in the surfactant is linearly related to its wettability, while the carboxyl group content is nonlinearly related to the wettability. The correlation equation of the surfactant wettability (contact angle) is as follows:

$$Z = A + BX$$  

(1.1)

where X is aliphatic (aromatic) methyl and Z is the contact angle.

After fitting, the following results can be obtained, as shown in Table 8:

4.2.2. Analysis of the Main Factors of Surfactant Wettability in Coal Samples with Medium and High Metamorphic Degrees. 4.2.2.1. Correlation Analysis between the Structural Parameters of Carbon and the Wettability of Medium and High Metamorphic Coal. The correlation analysis between the structural parameters of carbon and the wettability of medium and high metamorphic coal is as follows:

1. To analyze the main factors of surfactant wettability in nonstick coal samples.

The correlation analysis between the structural parameters of carbon in the surfactants and the wettability of the nonstick coal samples is shown in Table 9.

For the nonstick coal samples, the correlation between the lipid (aromatic) methyl group of the surfactant and the wettability is strong. Additionally, compared with the lignite samples, phenol or aromatic ether carbon shows a strong correlation with the wettability, both of which are significantly correlated with the wetting ability at the 0.05 level (both sides).

2. Analysis of the main factors of the wettability of the surfactant in the gas coal samples.

The correlation analysis between the structural parameters of carbon in the surfactants and the wettability of the gas coal samples is shown in Table 10.
In the gas coal sample, the affinity of aliphatic (aromatic) methyl, phenol, or aromatic ether carbon with the wettability is good. It is worth noting that the correlation between the nonprotonated carbon, bridging aromatic carbon, and oxygenated fatty carbon is strengthened in the gas coal sample.

Table 9. Correlation Analysis between the Structural Parameters of Carbon in the Surfactants and the Wettability of the Non-stick Coal Samples

| contact angle | aromatic carbon | carbonyl carbon | aromatic ring carbon | nonprotonated carbon |
|----------------|----------------|----------------|----------------------|----------------------|
| Pearson correlation | 1              | 0.080          | 0.134                | 0.090                |
| significance (bilateral) | 0.838          | 0.577          | 0.731                | 0.817                |

| contact angle | protonated carbon | phenol or aryl ether carbon | alkyl-substituted aromatic carbon | bridging aromatic carbon | fat–carbon ratio |
|----------------|-------------------|-----------------------------|---------------------------------|--------------------------|-----------------|
| Pearson correlation | 0.153            | −0.712                      | −0.204                          | 0.125                    | −0.298          |
| significance (bilateral) | 0.695            | 0.031                        | 0.599                           | 0.748                    | 0.436           |

| contact angle | ester (aromatic) methyl | quaternary carbon or methylene | oxygenated fatty carbon |
|----------------|--------------------------|--------------------------------|-------------------------|
| Pearson correlation | −0.734                  | 0.109                          | 0.123                   |
| significance (bilateral) | 0.024                    | 0.779                          | 0.752                   |

Table 10. Correlation Analysis between the Structural Parameters of Carbon in the Surfactants and the Wettability of the Gas Coal Samples

| contact angle | aromatic carbon | carbonyl carbon | aromatic ring carbon | nonprotonated carbon |
|----------------|----------------|----------------|----------------------|----------------------|
| Pearson correlation | 1              | −0.252          | 0.266                | −0.310               |
| significance (bilateral) | 0.513          | 0.490          | 0.416                | 0.267                |

| contact angle | protonated carbon | phenol or aryl ether carbon | alkyl-substituted aromatic carbon | bridging aromatic carbon | fat–carbon ratio |
|----------------|-------------------|-----------------------------|---------------------------------|--------------------------|-----------------|
| Pearson correlation | 0.146            | −0.635                      | −0.006                          | −0.387                   | −0.029          |
| significance (bilateral) | 0.707            | 0.066                        | 0.988                           | 0.304                    | 0.941           |

| contact angle | ester (aromatic) methyl | quaternary carbon or methylene | oxygenated fatty carbon |
|----------------|--------------------------|--------------------------------|-------------------------|
| Pearson correlation | −0.723                  | −0.075                        | 0.392                   |
| significance (bilateral) | 0.028                    | 0.849                         | 0.297                   |

Table 11. Correlation Analysis between the Structural Parameters of Carbon in the Surfactants and the Wettability of the Fat Coal Samples

| contact angle | aromatic carbon | carbonyl carbon | aromatic ring carbon | nonprotonated carbon |
|----------------|----------------|----------------|----------------------|----------------------|
| Pearson correlation | 1              | 0.095          | 0.139                | −0.034               |
| significance (bilateral) | 0.807          | 0.722          | 0.892                | 0.932                |

| contact angle | protonated carbon | phenol or aryl ether carbon | alkyl-substituted aromatic carbon | bridging aromatic carbon | fat–carbon ratio |
|----------------|-------------------|-----------------------------|---------------------------------|--------------------------|-----------------|
| Pearson correlation | 0.228            | −0.660                      | −0.350                          | 0.009                    | −0.216          |
| significance (bilateral) | 0.555            | 0.053                        | 0.356                           | 0.981                    | 0.576           |

| contact angle | ester (aromatic) methyl | quaternary carbon or methylene | oxygenated fatty carbon |
|----------------|--------------------------|--------------------------------|-------------------------|
| Pearson correlation | −0.716                  | 0.075                          | 0.143                   |
| significance (bilateral) | 0.030                    | 0.848                         | 0.714                   |

Table 12. Correlation Analysis between the Structural Parameters of Carbon in the Surfactants and the Wettability of the Coking Coal Samples

| contact angle | aromatic carbon | carbonyl carbon | aromatic ring carbon | nonprotonated carbon |
|----------------|----------------|----------------|----------------------|----------------------|
| Pearson correlation | 1              | 0.059          | 0.450                | −0.066               |
| significance (bilateral) | 0.881          | 0.224          | 0.866                | 0.964                |

| contact angle | protonated carbon | phenol or aryl ether carbon | alkyl-substituted aromatic carbon | bridging aromatic carbon | fat–carbon ratio |
|----------------|-------------------|-----------------------------|---------------------------------|--------------------------|-----------------|
| Pearson correlation | −0.140           | −0.835                      | −0.233                          | 0.025                    | −0.191          |
| significance (bilateral) | 0.719            | 0.005                        | 0.547                           | 0.950                    | 0.623           |

| contact angle | ester (aromatic) methyl | quaternary carbon or methylene | oxygenated fatty carbon |
|----------------|--------------------------|--------------------------------|-------------------------|
| Pearson correlation | −0.638                  | 0.238                          | −0.017                  |
| significance (bilateral) | 0.065                    | 0.537                         | 0.966                   |
The correlation analysis between the structural parameters of carbon in the surfactants and the wettability of the fat coal samples is shown in Table 11. In the fat coal sample, only the two carbon structures of lipid (aromatic) methyl and the wettability have a relatively close, with a correlation coefficient of 0.046 and a significant correlation at the level of 0.01 (both sides). Additionally, the correlation between lipid (aromatic) methyl and the wettability is relatively close, with a correlation coefficient of 0.065, which is slightly greater than 0.05.

(4) Analysis of the main factors of surfactant wettability in the coking coal samples

The correlation analysis between the structural parameters of carbon in the surfactants and the wettability of the coking coal samples is shown in Table 12. In the test with coking coal, phenol or aryl ether carbon has a very strong correlation with the wettability, with a significance coefficient of 0.005 and a significant correlation at the level of 0.01 (both sides). Additionally, the correlation between lipid (aromatic) methyl and the wettability is relatively close, with a correlation coefficient of 0.065, which is slightly greater than 0.05.

(5) Analysis of the main factors of surfactant wettability in the anthracite samples

The correlation analysis between the structural parameters of carbon in the surfactants and the wettability of the anthracite samples is shown in Table 13. In the test with the anthracite samples, only phenol or aryl ether carbon shows a strong correlation with wettability, with a correlation coefficient of 0.046 and a significant correlation at the level of 0.05 (both sides). The correlation between the carbon structure and the wettability of the other surfactants is weak.
In summary, in the correlation analysis between the carbon structure of the surfactants and the wettability, only phenol or arylether carbon and aliphatic (arylmethyl) methyl have a strong correlation with the wettability. Oxygen-bonded aliphatic carbon, carbonyl carbon, aromatic ring carbon, nonprotonated carbon, and alkyl-substituted aromatic carbon show certain correlations in some of the coal sample tests.

4.2.2. Construction of the Wettability Evaluation Model of the Structural Parameters of the Carbon in the Surfactants for Medium and High Metamorphic Coal. In the wettability test of the medium and high metamorphic grade coal samples, phenol or arylether carbon and lipid (arylmethyl) methyl in the surfactants are linearly related to their wettability. Therefore, the correlation equation between the surfactant wetting medium and high metamorphic grade coal (contact angle) is as follows

\[ Z = A + BX_1 + BX_2 \]  

(1.2) 

where \( X_1 \) is phenol or aryl ether carbon, \( X_2 \) is aliphatic (aromatic) methyl and \( Z \) is the contact angle.

The fitting results of the effects of the surfactant wettability parameters in medium and high metamorphic coal samples are shown in Table 14.

5. CONCLUSIONS

Based on the NMR experiments, the wetting mechanism between coal and the surfactants was studied, the key structural parameters affecting the wetting of coal by the surfactants were defined, and an evaluation model was established. The main conclusions are as follows.

1. In the process of coal evolution, the higher the rank of coal is, the higher the proportion of aromatic carbon is, and the lower the proportion of aliphatic carbon. All structural parameters basically conform to the law of carbon enrichment and deoxidation during coal metamorphism. The proportion of the aromatic carbon structure of the surfactants is lower, while the proportion of aliphatic carbon is higher.

2. Through \(^{13}\)C NMR experiments, the correlation between the structural parameters of the carbon of the surfactants and the wettability of the surfactants was obtained. It is proposed that only phenol or arylether carbon and aliphatic (arylmethyl) methyl have a strong correlation with wettable. Oxygen-bonded aliphatic carbon, carbonyl carbon, aromatic ring carbon, nonprotonated carbon, and alkyl-substituted aromatic carbon showed certain correlations in the testing of some coal samples, but the influence was small.

3. An evaluation model for the microstructure and wettability of surfactants with different coal species was established, and the contribution of phenol or arylether carbon and the fatty (arylmethyl) methyl in surfactants to the wettability of different coal species was quantitatively analyzed.

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Notes

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