Evaluation of the microwave-assisted organic acid separation of organic sulfur from high-sulfur coal

Jin Liu1 · Chaoshun Jiang1 · Hao Shen1 · Zheng Pang1 · Zhi Wang1 · Ping Li2 · Xing Xing3

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Abstract
To explore the feasibility of organic sulfur removal using organic acids, oxalic acid, citric acid and ascorbic acid were selected as desulfurization reagents. Combined with microwave irradiation technology, desulfurization experiments of high-sulfur coal treated with nitric acid were conducted. The orthogonal experimental results showed that the microwave synergistic citric acid desulfurization had a better desulfurization effect. The removal efficiency of organic sulfur was 55.47% when the operating conditions of the reaction were 1 mol/L, 90 °C and 1000 W for 7 min. The desulfurization efficiency of oxalic acid and ascorbic acid was low, and their organic sulfur removal efficiencies were 32.35% and 21.37%, respectively. Fourier transform infrared spectroscopy showed that sulfones and sulfoxides were partially reduced and the removal effect of thiophene was poor. X-ray photoelectron spectroscopy showed that the mercapto group in the mercaptan combined with hydrogen ions and escaped in the form of H2S. The content of aromatic thioether and aromatic thiol in the coal treated with microwave irradiation was only 0.1%. Research indicates that organic sulfur in coal can be easily reduced to sulfur-containing compounds under microwave heat and acid environment of citric acid. The outcomes provide helpful references for further studies on organic desulfurization reagents.

Graphical abstract
Keywords High sulfur coal · Coal desulfurization · Microwave irradiation · Organic acid

Introduction

With the development of China’s energy security strategy, high-sulfur coal will become a huge coal resource with potential economic value (Shen et al. 2021). Clean utilization of coal is a strategic choice for China’s low-carbon energy (Wang et al. 2019). It is extremely urgent to promote the development of clean energy and accelerate the development and utilization of clean coal from the entire life cycle and industrial chain (Sun et al. 2020), which is consistent with the global carbon dioxide neutrality strategy (Kawatra 2020). Coal clean technology enables us to curb the generation of harmful gases, such as SO₂ from the source, remove part of the ash and reduce the coal transportation cost, which is conducive to energy conservation and emission reduction and the circular development of the economy ( Çağlayan et al. 2021).

All types of inorganic sulfur in coal have significant differences in physical and chemical properties and are easy to remove (Takagi et al. 2004; Xu et al. 2015). However, the occurrence form of organic sulfur in coal is relatively complex, since coal is a sedimentary organic mineral with complex material composition and structure, which is transformed through the biochemical and physicochemical processes of plant remains. The sulfur atoms of organic sulfur are mostly connected with oxygen/hydrogen atoms in the form of chemical bonds, which are difficult to remove by ordinary desulfurization technology (Saikia et al. 2016). Research has shown that organic substances, such as phenol and nitrobenzene, can extract organic sulfur from coal (Liu et al. 2011). Some scholars have used chemical desulfurization technology (Cai et al. 2021) to show that the removal of S=S, S=O and -SH is the key to improving the removal efficiency of organic sulfur in high-sulfur coal (Tang et al. 2018), but utilizing of chemical desulfurization will reduce the calorific value. However, in current studies, researchers should attempt to maximize the removal efficiency of organic sulfur without reducing the calorific value of coal (Roy et al. 2021). Therefore, combined with the form of organic sulfur in coal, it is assumed that acidic organic matter can more easily react with organic matter in coal (Tang et al. 2021).

The application of microwaves makes the energy distribution of the desulfurization environment uniform, accelerates the dissolution of pyrite (Reddy et al. 2016), and protects the organic structure of coal under this mild heat (Mesroghli et al. 2015; Amjed et al. 2017). In this study, a new method of microwave-assisted organic acid removal of organic sulfur from coal was proposed to improve the desulfurization efficiency under the guarantee of coal quality. Three reducing organic acids (Fan et al. 2020) were selected as desulfurization solvents: oxalic acid, citric acid and ascorbic acid. Based on the single-factor experimental basis of optimal desulfurization of some organic acids (Liu et al. 2020), (i) the method of “microwave-assisted desulfurization of organic acids” was developed, (ii) the orthogonal scheme of desulfurization of organic acids was designed, and (iii) the optimal desulfurization conditions and organic sulfur removal mechanism were analyzed.

Materials and methods

Experimental materials

Raw coal characterization

Taking China’s high-sulfur coal as raw material, sampling techniques were similar to those of Jones riffles, and adapted coning and quartering methods (Mi et al. 2011). First, the superfine pulverized (Bokányi and Csöke, 2003) raw coal was put into the empty chamber to dry and screened with a standard sieve of 74 µm. The total sulfur content of the coal sample was determined by an automatic sulfur determination meter model (SDST20, accuracy of 0.03%). According to GB/T212-2008, an SDTGA408 moisture tester and a WS-G818 industrial analyzer were used for moisture measurement and industrial analysis. Sulfate and pyrite were determined according to GB/T215-2003 standard operation method. The Gross calorific value (GCV) of coal was determined by SDC715 according to GB/T213-2008 standard. The detailed procedures will be demonstrated in Sect. 2.3.

The pyrite and organic sulfur contents in the high-sulfur coal were 1.51% and 1.07%, respectively. The sulfur content in various forms in the coal was more evenly distributed with higher organic sulfur content, so it was suitable for desulfurization experimental research (Table 1). Characteristics of Coal B mentioned below are also in this table.

Organic acids

In our previous experiments, it was speculated that the acid additive might provide a suitable acidic environment for desulfurization due to the dissociated H⁺ during desulfurization. Thus, oxalic acid, citric acid and ascorbic acid were selected as desulfurizers by comparing the structure type and acidity degree of organic acids and combining the physicochemical properties of organic solvents (Liu et al. 2006; Mketo N et al. 2016), as shown in Table 2.
Table 1 Raw coal and Coal B of the characteristics

| Coal properties | Content |
|-----------------|---------|
|                 | Raw coal | Coal B  |
| Sulfur content  |          |         |
| Total sulfur    | 2.99%    | 1.17%   |
| Sulfate         | 0.41%    | 0.05%   |
| Pyrite          | 1.51%    | 0.08%   |
| Organic sulfur  | 1.07%    | 1.04%   |
| Industry analysis |        |         |
| Moisture        | 1.56%    | 1.14%   |
| Ash             | 11.84%   | 9.56%   |
| Volatile        | 10.36%   | 11.37%  |
| Fixed carbon    | 76.24%   | 77.93%  |
| Elemental analysis |       |         |
| Carbon          | 65.96%   | 67.71%  |
| Hydrogen        | 3.35%    | 4.16%   |
| Oxygen          | 3.26%    | 2.43%   |
| Nitrogen        | 1.72%    | 2.91%   |
| GCV             | Qgr, ad(J/g) | 29,972.46 | 29,657.47 |

Table 2 Organic acids to treat the coal

| Name               | Chemical formula | Structured | Structure type       | Acidity coefficient | References              |
|--------------------|------------------|------------|----------------------|---------------------|-------------------------|
| Oxalic acid        | C₂H₂O₄           | Binary carboxylic acid | 1.271 pKa | (Archambo et al. 2021; Liu et al. 2015) |
| Citric acid        | C₆H₈O₇           | Multiple carboxylic acid | 3.128 pKa | (Wei et al. 2021; Ekmekyapar et al. 2010) |
| Ascorbic acid      | C₆H₈O₆           | Aromatic acid | 4.040 pKa | (Zhu et al. 2019) |

Experimental process

In the early stage of the single-factor experiments, to avoid different factors affecting the desulfurization efficiencies in different combinations and levels, it was concluded that a series of optimal desulfurization parameter interval values should be divided into four-factor level interval values (Table 3) in the orthogonal experiment (orthogonal experimental design as Table 4) to more accurately find the optimal combination.

Microwave-assisted desulfurization of organic acids was conducted in a microwave reactor purchased from Sinusoidal Microwave Chemical Technology Co., Ltd. (Shanghai, China). The operating frequency was maintained at 2.45 GHz, and a schematic diagram of the microwave desulfurization reactor is shown in Fig. 1.

The experimental process is shown in Fig. 2. After crushing, the raw coal sample was screened out with a particle size of 58–74 μm to obtain coal sample. Coal sample was pickled to obtain coal B to avoid the effect of inorganic sulfur during the removal of organic sulfur. Then, 20 g of coal B was loaded into a three-port flask with 300 mL of organic additives, and the flask was put into the microwave extraction device to start the reaction according to the orthogonal experimental design. During the experiment, keep stirring. The exhaust gas was treated with NaOH solution; then, the desulfurized coal slime was filtered with a vacuum pump filter. The filtrate was repeatedly washed to neutral with deionized water. The coal was dried at 105 °C for 2 h and sealed after cooling. Through repeated experiments three times to get the average data to reduce the error caused by machines and personnel. Desulfurization efficiency and GCV were the evaluation basis. These operation steps were repeated for all three acids.
Data processing

In this study, the clean coal recovery efficiency, desulfurization efficiency, calorific value and calorific value loss efficiency are expressed by \( \eta_{/\%} \), \( R_{(S_{a,d})/\%} \), \( Q_{gr,ad} \) and \( L_{Q} \), respectively. The results are all accurate to the percentile. Neutralization symbol of the calculation formula is shown in Table 5.

![Fig. 1](image_url)

Table 3 Desulfurization condition factor levels

| The factor | Value range |
|-----------|-------------|
| A: Concentration of organic acid (mol/L) | 0.1 0.2 0.5 1 |
| B: Reaction temperature (°C) | 70 80 90 100 |
| C: Reaction time (min) | 5 7 9 11 |
| D: Microwave power (W) | 700 800 900 1000 |

Table 4 Orthogonal experimental design scheme

| Serial number | A (Solution concentration) | B (Reaction temperature) | C (Reaction time) | D (Microwave power) |
|---------------|----------------------------|--------------------------|-------------------|---------------------|
|               | C/(mol/L)                  | T/(°C)                   | t/(min)           | P/(W)               |
| L₁            | 0.1                        | 70                       | 5                 | 700                 |
| L₂            | 0.1                        | 80                       | 7                 | 800                 |
| L₃            | 0.1                        | 90                       | 9                 | 900                 |
| L₄            | 0.1                        | 100                      | 11                | 1000                |
| L₅            | 0.2                        | 70                       | 7                 | 900                 |
| L₆            | 0.2                        | 80                       | 5                 | 1000                |
| L₇            | 0.2                        | 90                       | 11                | 700                 |
| L₈            | 0.2                        | 100                      | 9                 | 800                 |
| L₉            | 0.5                        | 70                       | 9                 | 1000                |
| L₁₀           | 0.5                        | 80                       | 11                | 900                 |
| L₁₁           | 0.5                        | 90                       | 5                 | 800                 |
| L₁₂           | 0.5                        | 100                      | 7                 | 700                 |
| L₁₃           | 1                          | 70                       | 11                | 800                 |
| L₁₄           | 1                          | 80                       | 9                 | 700                 |
| L₁₅           | 1                          | 90                       | 7                 | 1000                |
| L₁₆           | 1                          | 100                      | 5                 | 900                 |
Microwave-assisted organic acid desulfurization results

Orthogonal experimental results and analysis

Through the orthogonal test of 4 factors and 4 levels, the determination results of the three organic acids at different concentrations are shown in Tables 6, 7 and 8.

Desulfurization efficiency and calorific value loss efficiency of coal were taken as evaluation indexes. The highest desulfurization efficiency was selected as the optimal desulfurization combination. If the desulfurization rate difference between the two groups was less than 2%, the highest calorific value was selected as the optimal group. The optimal group was selected from the experimental results of each acid (Table 9). Citric acid can reduce the organic sulfur content to 0.48%, which has a better effect than the performance in Table 1.
Summary of the best experimental conditions

The sulfur removal efficiency and calorific value loss efficiency were calculated using the experimental results. The best desulfurization effect was obtained when the citric acid solution with a concentration of 1 mol/L and the coal sample with a particle size of 58 ~ 74 μm reacted for 7 min at 90 °C and 1000 W microwave radiation. Microwave-assisted citric acid desulfurization (MACAD) had a good effect on the organic sulfur removal, which reduced the total sulfur content from 1.17 to 0.63 and the organic sulfur content from 1.05 to 0.48.

Citric acid can effectively remove organic sulfur from high sulfur coal, and the removal efficiency was 55.47%. The other two have the disadvantages of low desulfurization efficiency and lower coal calorific value (Fig. 3).

Table 8 Results of the ascorbic acid desulfurization experiment

| Serial number | R(%) | S,do (%) | R_{St,d}(%) (%) | Q_{gr,ad}(J/g) | LQ (%) |
|---------------|------|----------|-----------------|----------------|--------|
| K-1           | 98.25 | 1.13 | 5.11 | 28,519.77 | 3.82 |
| K-2           | 96.50 | 1.07 | 11.75 | 28,691.21 | 3.25 |
| K-3           | 96.75 | 1.05 | 13.17 | 28,890.79 | 2.57 |
| K-4           | 96.00 | 0.99 | 18.77 | 28,666.96 | 3.33 |
| K-5           | 95.90 | 1.09 | 10.66 | 28,482.64 | 3.95 |
| K-6           | 96.55 | 1.05 | 13.35 | 28,532.37 | 3.78 |
| K-7           | 95.55 | 1.10 | 10.17 | 28,711.32 | 3.18 |
| K-8           | 95.80 | 1.06 | 13.21 | 28,762.05 | 3.01 |
| K-9           | 96.50 | 1.08 | 10.92 | 28,652.32 | 3.38 |
| K-10          | 96.35 | 1.07 | 11.89 | 28,411.66 | 4.19 |
| K-11          | 94.10 | 1.09 | 12.33 | 28,661.35 | 3.35 |
| K-12          | 95.50 | 1.09 | 11.03 | 28,649.37 | 3.39 |
| K-13          | 94.85 | 1.11 | 10.01 | 28,665.44 | 3.33 |
| K-14          | 95.00 | 1.11 | 9.87  | 28,680.42 | 3.28 |
| K-15          | 95.80 | 1.03 | 15.66 | 28,756.91 | 3.02 |
| K-16          | 96.25 | 1.05 | 13.62 | 28,722.06 | 3.15 |

Table 9 Results of the optimal organic acid desulfurization group

| Organic acid | Concentration (mol/L) | Reaction temperature (°C) | Reaction time (min) | Microwave power (W) | Inorganic sulfur content (%) | Organic sulfur content (%) | Total sulfur content (%) |
|--------------|-----------------------|----------------------------|---------------------|---------------------|-----------------------------|--------------------------|------------------------|
| Oxalic acid  | 1                     | 80                         | 9                   | 700                 | 0.12                        | 0.73                     | 0.85                   |
| Citric acid  | 1                     | 90                         | 7                   | 1000                | 0.15                        | 0.48                     | 0.63                   |
| Ascorbic acid| 0.1                   | 100                        | 11                  | 1000                | 0.13                        | 0.86                     | 0.99                   |
to sulfoxide or even sulfide. When the wavenumber was approximately 1033.7 cm⁻¹, the absorption peak intensity was significantly reduced, which corresponded to the stretching vibration of S=O of sulfoxide (R₁-SO-R₂). This behavior can be attributed to the citric acid solution extracting the sulfoxide for desulfurization. The wavenumber at 538.1 cm⁻¹ was the absorption peak of aromatic thioether, and the absorption peak intensity was weakened compared with that before desulfurization, which indicates that the aromatic thioether was removed. This analysis is consistent with the X-ray photoelectron spectroscopy (XPS) analysis in the following section.

In summary, FT-IR analysis shows that the desulfurization process only affects a small part of the sulfur-containing groups in pyrite and has no effect on the macromolecular structure of coal itself.

**X-ray photoelectron energy diffraction analysis**

The experimental results show that citric acid is an effective desulfurizer. To further explore the reasons for the high desulfurization rate, XPS was used to analyze the surface chemical states of coal samples before and after the desulfurization of citric acid. Qualitative and semiquantitative analyses were mainly conducted on the valence state and sulfur element content in the coal to explore the underlying mechanism.

The photoelectron binding energy varied according to different forms of sulfur in the coal sample (iron sulfide: 161.3 ~ 162.2 eV; mercaptan and sulfide: 162.2 ~ 163.2 eV; sulfoxones: 164.1 ~ 164.4 eV; sulfone: 165.0 ~ 167.0 eV; thiophene: 167.0 ~ 169.0 eV) (Ma et al. 2014). Under the excitation of X-rays, electrons in the sulfur 2p layer will produce energy level splitting, and the energy correction of S2p is performed through surface pollution of C 1s (284.5 eV) as the standard. XPS analysis (Fig. 5 and Fig. 6) was conducted on the coal samples, and peak fitting was conducted to obtain the contents of different sulfur forms in the coal samples (Table 10).

According to the analysis, the effect of the MACAD method was ideal: sulfoxide and sulfonate were significantly removed. The thiophene removal effect was not ideal, possibly due to the penta-heterocycles, and the structure of thiophene was relatively stable and difficult to remove. The contents of mercaptan and sulfide were almost completely removed from 0.14% to 0.01%. Simultaneously, sulfoxide and sulfonate organic sulfur can be separated by citric acid extraction (Smith et al. 2001).

**Analysis of the desulfurization mechanism of citric acid**

Under the microwave-assisted action, due to the strong reductivity of citric acid, the S=O bond expands and vibrates, which reduced sulfoxones and sulfides to sulfides, as shown in Fig. 7. In addition, due to the difference in physical and chemical properties between citric acid solution, sulfoxides and sulfonic acids, desulfurization can be achieved by extraction, as shown in Fig. 8 (Jin et al. 1989; Nishida K et al. 2017). Aromatic mercaptan is mainly subjected to microwave irradiation to dissociate the sulphydryl
group; then, free radicals bind to $\text{H}^+$ and escape in the form of $\text{H}_2\text{S}$, as shown in Fig. 9 (Mu et al. 2021). The C-S bond of aromatic thioether was broken by microwave thermal irradiation, and the sulfur-containing groups were converted to $\text{SO}_2^-$, as shown in Fig. 10 (Xu et al. 2015). The C-S bond

![Fig. 5 Pictorial diagram and waterfall plot of the X-ray photoelectron diffraction analysis (the sample targeted was coal sample B)](image1)

![Fig. 6 Pictorial diagram and waterfall plot of the X-ray photoelectron diffraction analysis (the sample was clean coal desulfurized by citric acid)](image2)

| Table 10 | Comparison table of the sulfur content before and after desulfurization |
|----------|-------------------------------------------------------------|
| Raw coal | Optimum desulfurization of coal |
| Peak points | Form of sulfur | Content (%) | Peak points | Form of sulfur | Content (%) |
| 1 | Thiophene | 0.44 | 1 | Thiophene | 0.31 |
| 2 | Sulfoxide | 0.27 | 2 | Sulfoxide | 0.11 |
| 3 | Sulfones | 0.20 | 3 | Sulfones | 0.05 |
| 4 | Mercaptan and sulfide | 0.14 | 4 | Mercaptan and sulfide | 0.01 |
in thiophene is almost unbroken because of its five-membered heterocyclic structure.

**MACAD calorific value analysis**

The desulfurization effect can be evaluated via the calorific value of coal. Table 11 shows the calorific values of B-coal and clean-coal. After the MACAD treatment, GCV of coal samples decreased, which may be due to the unstable chemical bond fracture of organic matter under the action of microwave irradiation, which caused the structural change in coal samples. However, the extent of its decline indicates that the structural change was not great. By referring to the international standard *BS ISO 11760–2018 Classification of coals* and the Chinese standard *GB/T15224.3–2004*, coal remains in the grade of extra high calorific value coal after the pickling treatment. It has certain reference value to the coal washing industry.

**Comparison of the results**

The researchers desulfurized the sample under the microwave irradiation. After the treatment, most of FeS₂ was transferred, while -SH and S–S required more time to rupture than S = O when the microwave power was 1000 W (Wu et al. 2019). Gürüş oxidized pulverized coal through nitric acid solution, and the conversion rate was only 38.7% (Gürüş et al. 2007). Compared with previous chemical desulfurization research reagents, such as hydrogen peroxide, sodium hydroxide, nitric acid, and peracetic acid (Xia et al. 2018; Wang et al. 2021), MACAD achieves desulfurization in multiple ways. Sulfones, sulfoxides, mercaptans and thioether can be effectively removed by microwaves and citric acid. Citric acid also has the advantage of extractable organic sulfur over other chemical additives. In addition, the traditional chemical desulfurization method may destroy the molecular structure of coal, but this method can avoid this side effect.

**Conclusion**

The study shows that it is feasible to remove sulfur from coal by organic acids, especially organic sulfur. Microwave citric acid desulfurization is a promising coal washing technology.
Using citric acid to provide low concentration hydrogen ion environment can not only achieve the desulfurization effect, but also ensure that the macromolecular structure of coal is not destroyed. Under the heat energy provided by microwave, it is further enhanced. This paper is expected to support other follow-up studies. The summary and prospect of this study are as follows: (1) Other organic acid desulfurization experiments should be further explored to find more efficient organic desulfurization reagents than citric acid. (2) When exploring the mechanism of microwave-assisted desulfurization, the dielectric constants of ash impurities and sulfur in coal can be used to demonstrate.

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Data availability All data reported in the study are available from the authors.

Declarations

Conflict of interest The authors have not disclosed any competing interests.

Ethical approval The data came from the team’s experimental study.

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Authors and Affiliations

Jin Liu1 · Chaoshun Jiang1 · Hao Shen1 · Zheng Pang1 · Zhi Wang1 · Ping Li2 · Xing Xing3

1 College of Safety Engineering, Chongqing University of Science & Technology, Chongqing 401331, China
2 Chongqing Energy Investment Group Co. Ltd, Chongqing 40121, China
3 School of Materials Science and Engineering, UNSW Sydney, Kensington, NSW 2052, Australia