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Effect of short-term weathering on kaolin preparation from coal gangue via flotation decarburization

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Abstract: Coal-series kaolin flotation decarburization efficiency is greatly influenced by weathering. This study investigated the mechanisms by which three typical types of weathering (short-term natural weathering, low-temperature oxidation and freeze–thaw weathering) affect the surface characteristics and floatability of low-rank coal. Zeta potential measurements, scanning electron microscopy (SEM), Fourier transform infrared (FTIR) spectroscopy and contact angle analysis were performed to investigate these mechanisms. Results revealed the following: (1) after 20 months of natural weathering, cracks formed on the coal surface, accompanied by the gradual disappearance of $-CH_3$ and –C–H functional groups. Continued weathering increased the ash content, reduced hydrophobicity and deteriorated floatability. (2) Freeze–thaw weathering for 60 days had a minimal impact on coal hydrophobicity and flotation performance, with only slight fluctuations observed in the flotation yield and ash content of the recovered coal. (3) Low-temperature oxidation over 15 days induced crack development, particle size refinement and complete disappearance of hydrophobic $-CH_2$ and –C–H functional groups, leading to severe deterioration in floatability. Thus, this study confirms that low-temperature oxidation exerts the most pronounced negative impact on flotation decarburization efficiency.

Keywords: flotation, low-rank coal, coal gangue, natural weathering, low-temperature oxidation

1. Introduction

Kaolin is an essential non-metallic mineral resource and raw material for materials such as ceramics, papers and coatings (Hady et al., 2024). China has abundant and widely distributed kaolin reserves, of which coal-bearing kaolin accounts for approximately 50%. Coal-based kaolin has low whiteness as it contains traces of residual coal and organic carbon; therefore, it is pre-treated via high-temperature calcination, re-election or flotation to reduce the carbon content (Chen et al., 2024, Nguyen et al., 2024, Fu et al., 2018, Wang et al., 2024). At present, carbonaceous impurities are mainly removed via roasting, which considerably improves the whiteness of kaolin. However, this method consumes large amounts of energy and causes environmental pollution. Simeng (Simeng et al., 2022) proposed a kinetic method for decarburising calcined coal-based kaolin that achieved a high decarburization rate of 99.9%, whiteness up to 89. 3% and an oil absorption value of 76.1 g/100 g after calcining at 900°C for approximately 3.3 s. Gravity separation is another method used for impurity separation, which is mainly based on the difference in settling rates of organic carbon and coal-series kaolin with different densities. As quartz, pyrite, and other impurities have the same density as kaolin, they cannot be efficiently separated (Hou et al., 2024, Xia, 2016). Therefore, gravity separation is often used in conjunction with other methods such as magnetic separation and leaching.

Raw kaolin ores containing higher amounts of impurities are treated via flotation, and impurities such as organic carbon, iron, and titanium are removed via multistage flotation. Ren (Ren et al., 2023) suggested that collector containing ester groups and long carbon chains (carbon atoms between 13 and 15) are effective for de-carbonising coal-series kaolin. Huang (Huang et al., 2024) reported that weak

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alkaline and acidic environments are ideal for the flotation of iron-bearing minerals such as mica from quartz and feldspar. Barani (Barani and Kalantari, 2018) demonstrated that cetylpyridinium chloride (CPCI) and dodecylamine (DDA) were the best trapping agents under acidic conditions, offering the best selectivity for kaolin. Flotation can considerably improve the whiteness of kaolin; however, the use of reagents increases the production costs and causes environmental pollution (Fan et al., 2025).

Coal-series kaolin is a by-product of coal development and utilisation and a solid waste that is generated in large quantities and discharged. Coal gangue is often piled up in the open air, exposed to the air for a long time, and its structure is gradually destroyed. The large temperature difference between day and night accelerates the change of its physical form, and the freezing and thawing effect further aggravates the crushing of coal gangue. In the dry environment, the gangue is very easy to occur low-temperature oxidation reaction, its internal combustible components in the slow oxidation process continues to accumulate heat, reach a certain temperature, it will trigger spontaneous combustion. Spontaneous combustion of coal gangue will not only release a large amount of harmful gases, polluting the surrounding atmospheric environment, but also cause damage to the ecosystem and soil structure around the gangue mountain, bringing serious environmental problems. The minerals in coal-series kaolin undergo physicochemical changes due to several environmental factors such as temperature and moisture, thereby impacting flotation. Yuchu Cai (Cai et al., 2019b) reported that accumulated coal slurry oxidises, thereby increasing the O content and decreasing the C and H contents. As a result, the corresponding numbers of oxygen-containing functional groups such as C-O-C, C=O and O=C-O and other polar functional groups increase, making the coal surface more hydrophilic. This hydrophilic surface hinders bubble mineralisation, thereby hampering the flotation efficiency. To address these issues, optimal flotation conditions were proposed: impeller speed: 1,800 r/min; slurry concentration: 60 g/L and air flow rate: 0.25 m3/(m2-min). Due to water evaporation, the weak hydroxyl bond fractured and was consumed during methylene reaction, generating carboxylic acid. The aliphatic hydrocarbons considerably increase due to the impact of oxygen-containing functional groups in C=O, enhancing the hydrophilicity of minerals as well as impacting gangue flotation (Yu et al., 2025).

Coal-series kaolin is co-associated with long-flame coal, non-sticky (sticky) coal and other coals with low metamorphic degree. It has large surface porosity and high oxygen content and contains abundant hydrophilic functional groups on the surface. However, the influence of kaolin weathering on its floatability has been scarcely studied. Therefore, the effect of short-term natural weathering, low-temperature oxidation, and freeze–thaw weathering on the recovery of low-rank coal in coal-series kaolin by flotation was investigated herein using approaches such as zeta potential test, scanning electron microscopy (SEM), Fourier transform infrared (FTIR) spectroscopy, and contact angle measurements. Moreover, the influence mechanism of weathering on the decarbonization and floatability of coal-series kaolin was reported.

2. Material and methods

2.1. Experimental materials

Coal samples were obtained from a coal preparation plant in Jungar Coalfield, China. To quantify the study object, handpicked kaolin was blended with refined coal in a 3:1 ratio to obtain a synthetic gangue. This area experiences mid-temperate continental climate and is close to the desert; therefore, it receives low precipitation and the day and night temperatures considerably vary (Lu et al., 2024b). Table 1 and Fig. 1 show the chemical and physical phase compositions of coal-series kaolin. The silica–aluminium ratio in coal-series kaolin was approximately 1.12, with high purity of kaolin (Lv et al., 2024). Long-flame coal was found in the study site; its industrial and elemental analyses are shown in Table 2. This coal contained abundant C and O and hydrophilic groups, with a high oxidation degree; these factors considerably impacted its flotation.

Table 1. Chemical composition analysis of kaolin

Ingredient	Al_2O_3	SiO_2	Fe_2O_3	TiO_2	SO_3	loss-on-ignition
Content/%	44.856	44.856	1.39	1.301	0.689	15.56

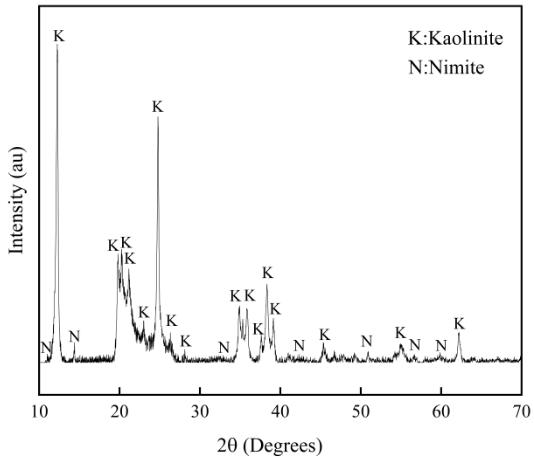

Fig. 1. XRD spectra of kaolin

Table 2. Industrial and elemental analysis of coal

M_{ad} (%)	A_d (%)	V_{daf} (%)	C_{daf} (%)	H_{daf} (%)	O_{daf} (%)	N_{daf} (%)	S_{td} (%)
2.55	20.46	20.29	65.11	3.81	12.09	1.05	0.48

Kaolin has relatively stable physicochemical properties. Herein, the apparent effects of natural weathering, freeze–thaw weathering, and low-temperature oxidation on flotation were studied mainly for low-rank coals (Tan et al., 2016). It was crushed using a jaw crusher EP-1 and screened to approximately 0.075 mm using a circular vibrating screen and then placed in an outdoor environment for weathering. The samples after weathering treatment for 0, 4, 6, 15, and 20 months were selected. Freeze–thaw weathered samples were placed at −20°C ± 1°C for 12 h, followed by 40°C ± 1°C for 12 h. These conditions were maintained in a cycle test, and the specimens after 10, 20, 30, 40, 50, and 60 days of tests were selected. The coal samples were subjected to low-temperature oxidation in an electrically heated blast drying oven at 80°C ± 1°C for 0, 1, 2, 4, 8, and 15 days.

2.2. Experimental methods

This study conducted FTIR, SEM, zeta potential, contact angle measurements, and flotation tests to investigate the effects of natural weathering, freeze-thaw cycles, and low-temperature oxidation on the flotation decarburization efficiency for kaolin recovery from low-rank coal. Detailed experimental procedures are illustrated in Fig. 2.

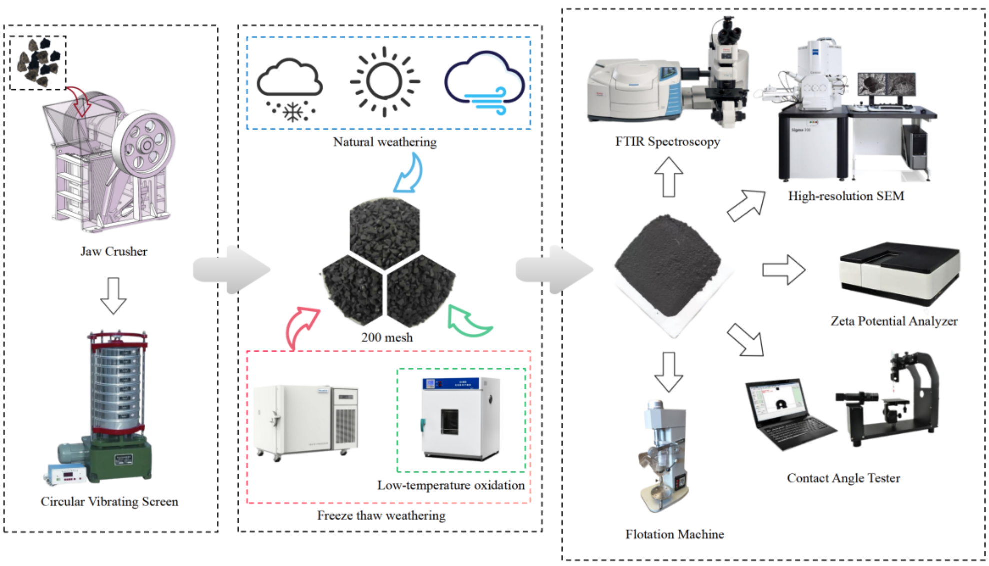

Fig. 2. Roadmap for the experiment

2.2.1. FTIR tests

The functional group variations in coal samples were analyzed using an IRTracer-100 Fourier transform infrared spectrometer (FTIR). The distribution of functional groups in low-rank coal was characterized via the potassium bromide (KBr) pellet method. The acquired FTIR spectra were processed and analyzed using Origin software.

2.2.2. SEM tests

The surface coatings of low-rank coal particles were analysed via high-resolution SEM (MAIA3 LMH, Tescan, Czech Republic).

2.2.3. Zeta potential tests

The electrochemical properties of low-rank coal were analyzed using a JS94H2M electrochemical workstation (Shanghai Zhongchen Co., Ltd.). To ensure accuracy, the experiments were conducted in 10 replicates, and the arithmetic mean of the measurements was calculated.

2.2.4. Contact angle tests

Analyses were carried out with a contact angle goniometer (Li-Chen Scientific Instruments Co., Ltd.). The hydrophilicity of naturally weathered, freeze-thaw-weathered, and low-temperature-oxidized low-rank coals was assessed via the sessile drop method with distilled water. To ensure reliability, triplicate measurements were performed, and the arithmetic mean was calculated and reported as the final value.

2.2.5. Laboratory flotation tests

Flotation test was performed in a 1.5 L flotation cell with an impeller speed of 1,870 r/min and solid concentration of 66.67 g/L. Sec-octanol (200 g/t) (Pawliszak et al., 2024 , Wang et al., 2023) and diesel fuel (1,000 g/t) (Gredelj et al., 2008) were used as the frother and collector, respectively. The detailed process is shown in Fig. 3. two reverse flotation tests were carried out and the results are the arithmetic mean.

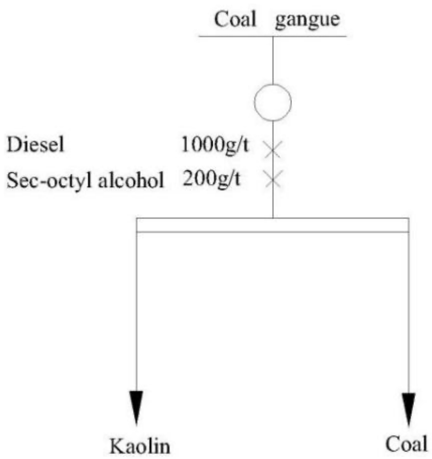

Fig. 3. Flotation test flow chart

Herein, reverse flotation performance was assessed in terms of the concentrate yield, which was calculated using Equations (1) and (2).

$$\gamma_{flo} = \frac{m_{flo}}{m_{feed}} \times 100\% \quad (1)$$

$$\gamma_{sed} = \frac{m_{sed}}{m_{feed}} \times 100\% \quad (2)$$

where γ_{flo} is the flotation yield (%), m_{flo} is the float mass (kg), γ_{sed} is the sediment yield (%), m_{sed} is the sediment mass (kg) and m_{feed} is feed quality (kg).

3. Results and discussion

3.1. Effect of natural weathering on kaolin preparation via flotation decarburization

3.1.1. FTIR spectroscopy

FTIR spectroscopy was performed on low-rank coals with different outdoor natural weathering times. The corresponding results are shown in Fig. 4. The spectra showed -CH, $-CH_3$ and C-H vibration peaks in unweathered samples. These absorption peaks gradually decreased and disappeared in naturally weathered with respect to time. Unweathered coal samples contained less hydrophilic oxygen content and more hydrophobic hydrocarbon groups (Sabereh et al., 2023). In the process of natural weathering, some clay minerals in the gangue, the crystal lattice structure will be gradually destroyed, the destruction of the crystal structure leads to changes in the chemical environment in which -OH, C-I, water is reduced, so the original regular arrangement of -OH, C-I becomes disordered, resulting in -OH, C-I absorption peaks become broader, weaker, or even no longer clear (Cheng et al., 2024). As the natural weathering continued, the hydrophobic groups gradually decreased and disappeared, thereby gradually decreasing the hydrophobicity of naturally weathered samples; these factors result in the poor floatability of low-rank coals.

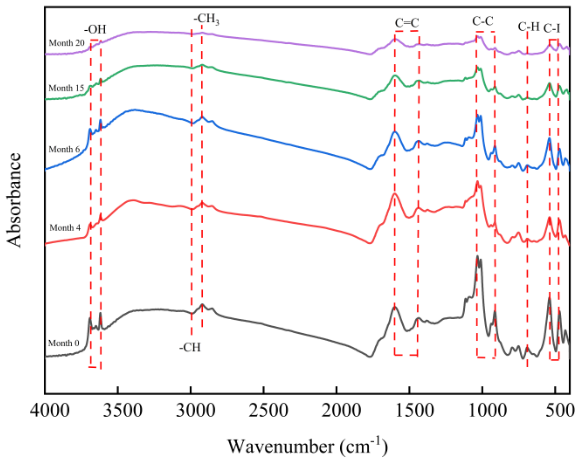

Fig. 4. FTIR of coal samples with different natural weathering times

3.1.2. SEM

Micro-morphological analysis was performed on low-rank coal samples naturally weathered for different times, and the corresponding results are shown in Fig. 5. The surface of fresh coal samples was dense and had good integrity. As the weathering time increased, the surface coarsened, producing line cracks and grooves as well as hollow and small lumps of particles (Guo et al., 2016). This indicated surface cracking due to weathering, which formed fine particles that deteriorated their floatability (Lu et al., 2024a). Outdoor weathering oxidised the organic matter on the surface of the coal samples, resulting in their scale-like peeling and destroying the lattice structure of coal.

3.1.3. Zeta potential tests

Zeta potential tests showed that as the outdoor natural weathering time increased, the flotation recovery of low-rank coals gradually deteriorated. Moreover, the absolute zeta potentials gradually became more negative from −91.5 mV to −103.14 mV as the weathering time increased from 4 months to 6 months and ultimately reached a plateau (Fig. 6). The microscopic particle rejection of coal samples gradually increased due to natural weathering, thereby deteriorating their floatability and yield. These findings further validated the SEM results.

3.1.4. Contact angle measurements

The contact angles of coal samples naturally weathered for different times were measured. Results revealed that the fresh coal samples had the largest contact angle of approximately 52.30° and decreased to 43.19° after 20 months of outdoor natural weathering. As shown in Fig. 7, this reduction in the contact angle indicates a decrease in the contact angle of the low-rank coal that results in a deterioation of its floatability (Mateusz et al., 2024).

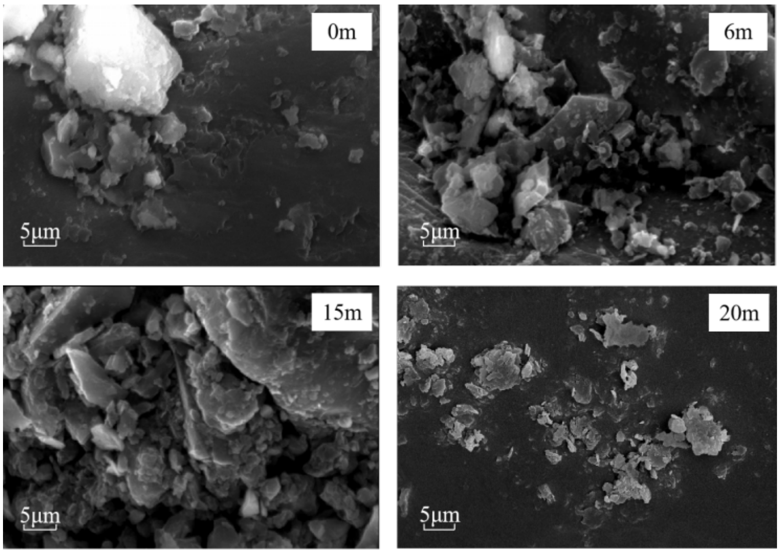

Fig. 5. Scanning electron microscopy (SEM 20,000x) of coal samples with different natural weathering times

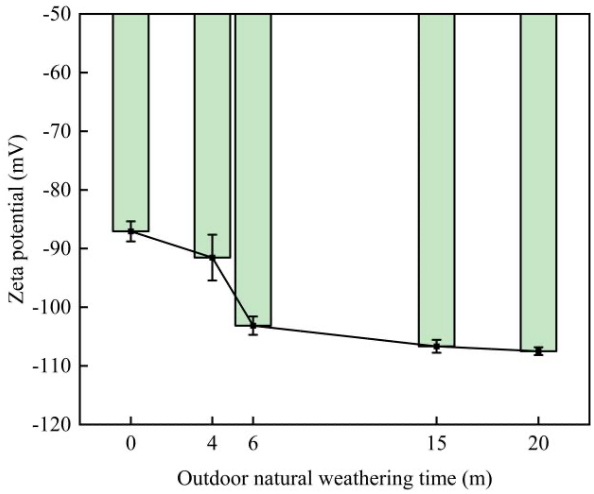

Fig. 6. Zeta potential variation of coal samples with different natural weathering times

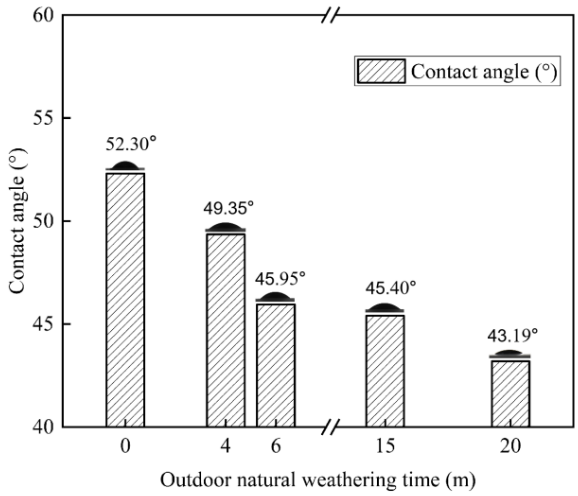

Fig. 7. Contact angle variation of coal samples with different natural weathering times

3.1.5. Laboratory flotation tests

Under prolonged natural weathering, float production decreased sharply in the first six months, and as time passed, float production (Fig. 8). The ash content of the unweathered low-rank was 20.74%, which increased to 38.50% after 20 months (Fig. 9), because the reactive groups in the side chains of the macromolecular structural units of organic matter in coal samples adsorbed atmospheric oxygen and formed coal–oxygen complexes (Lv et al., 2024). As natural weathering continued, these complexes decomposed and released carbon dioxide, carbon monoxide, water and heat (Science et al., 2019). This reduced the floatability of low-rank coal coals, conforming with the aforementioned test results.

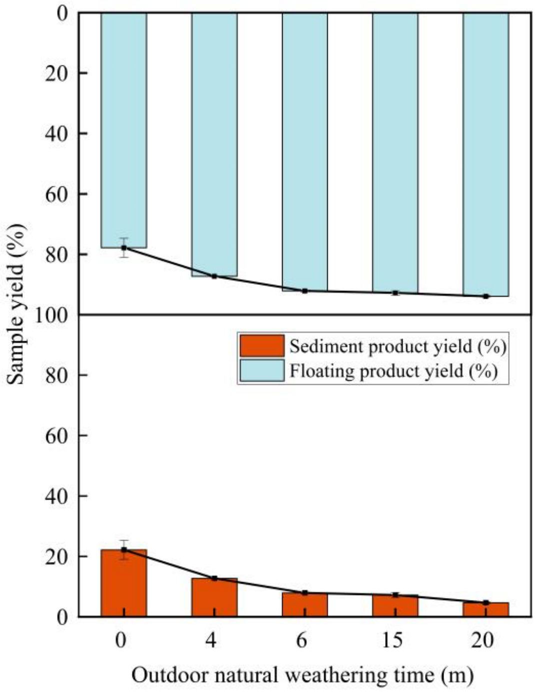

Fig. 8. Variation of yield of coal samples with different natural weathering times

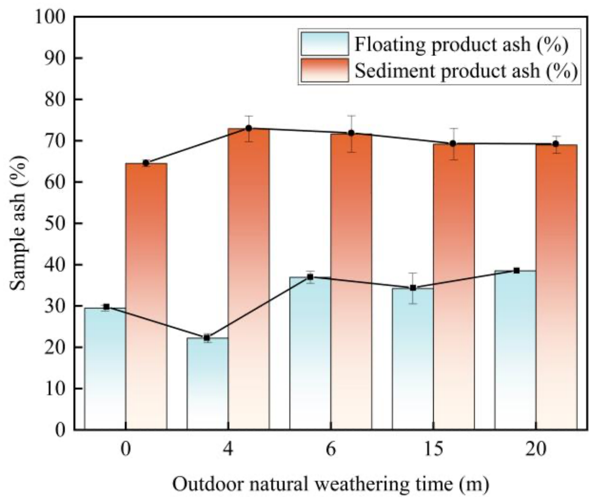

Fig. 9. Variation of ash content of coal samples with different natural weathering times

3.2. Effect of freeze-thaw weathering on kaolin preparation via flotation decarburization

3.2.1. FTIR spectroscopy

Fig. 10 shows the FTIR spectra of the coal samples subjected to freeze–thaw weathering for varying durations. Distinct absorption bands corresponding to –CH/–CH_2 deformation vibrations and –C–H out-of-plane bending modes (Kai et al., 2022) were observed in the spectra of the unweathered coal samples. Notably, these characteristic peaks remained largely unchanged with increasing freeze–thaw cycles, implying there were minimal changes in surface hydrophobicity. Consequently, negligible deterioration was observed in the floatability of coal particles under freeze–thaw conditions, suggesting that such weathering marginally impacted the recovery efficiency of low-rank coal from coal-series kaolin (Xiao et al., 2024).

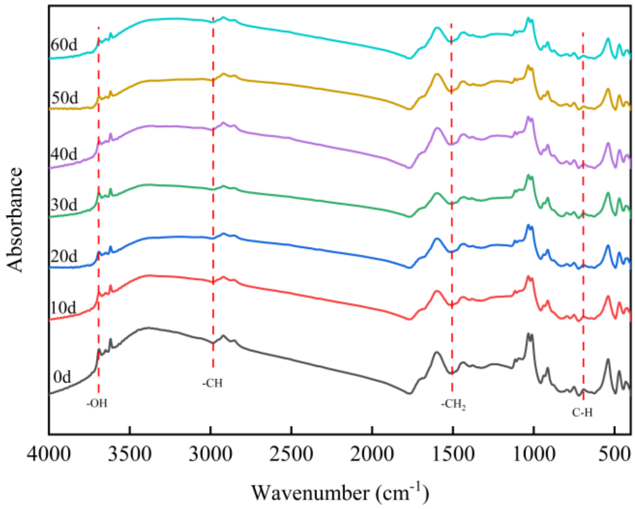

Fig. 10. FTIR of coal samples with different freeze-thaw weathering times

3.2.2. SEM

Fig. 11 shows the SEM images of the coal samples exposed to varying freeze–thaw cycles. Microfractures progressively developed on the coal surface with prolonged weathering duration (Zhao et al., 2018; Cheng et al., 2023). Notably, short-term (60 days) freeze–thaw treatment induced only limited structural damage, correlating with marginal alterations observed in particle floatability.

3.2.3. Zeta potential tests

Increasing the number of freeze-thaw cycles had little effect on coal, which was mainly concentrated at 90 mV (Fig. 12). The zeta potentials changed only slightly and exhibited a stable state. These findings indicated that freeze–thaw weathering did not impact the flotation recovery of low-rank coals considerably.

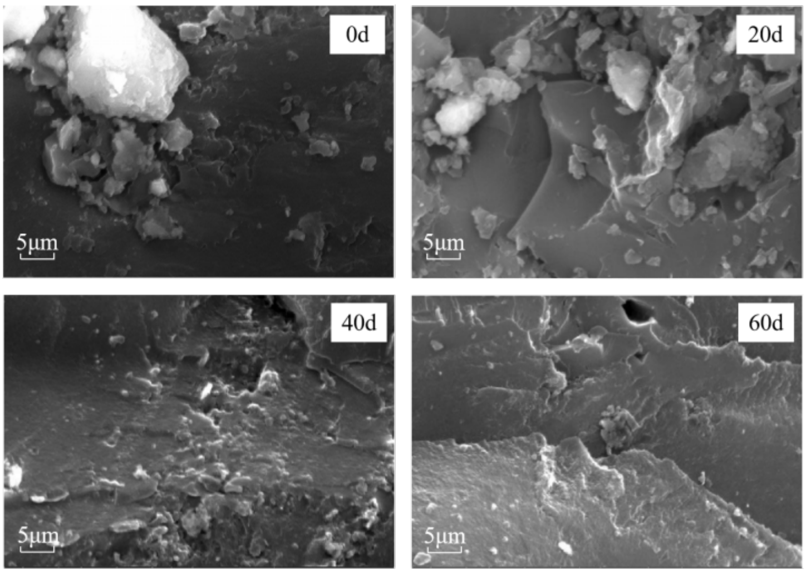

Fig. 11. Scanning electron microscopy (SEM 20,000x) of coal samples with different freeze-thaw weathering times

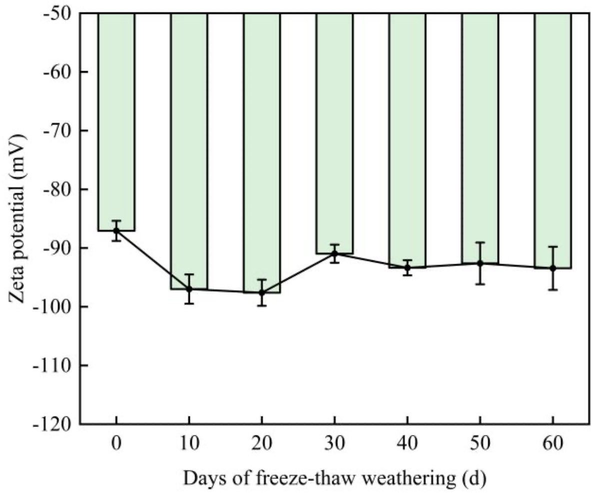

Fig. 12. Zeta potential variation of coal samples with different freeze-thaw weathering times

3.2.4. Contact angle measurements

Fig. 13 shows that the contact angles of coal samples at freeze–thaw oxidisation times of 0–60 days fluctuates between 45° and 53°, with a small change trend mainly concentrated at approximately 50°. As freeze–thaw weathering continued, the hydrophilicity of the coal sample surface did little change considerably (Xue et al., 2025). This indicated that the flotation of low-rank coal was not considerably impacted.

3.2.5. Laboratory flotation tests

After subjecting the coal samples to different freeze–thaw cycles for flotation test, the sink yield of low-rank coals was 70%–80%. This yield did not change considerably with the number of cycles, as shown in Fig. 14. The ash content of sinkary low-rank coal first increased and then decreased (Fig. 15). Results indicated that freeze–thaw weathering only slightly impacted the recovery of low-rank coal via flotation.

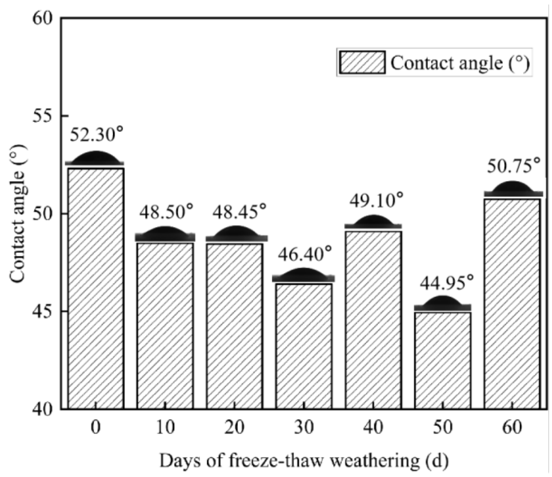

Fig. 13. Contact angle variation of coal samples with different freeze-thaw weathering times

3.3. Effect of low-temperature oxidation on kaolin preparation via flotation decarburization

3.3.1. FTIR spectroscopy

Fig. 16 shows the FTIR spectra of low-rank coal samples with different low-temperature oxidation times. The spectra show peaks corresponding to the deformation vibration of -CH_2 on the alkane chain structure and the out-of-plane bending vibration of -C-H on the aryl nucleus. As the low-temperature oxidation time increased, these absorption peaks considerably decreased and disappeared completely

(Hao et al., 2024). The C-H and C-O groups in coal oxidised at low temperatures and the water within the coal particles evaporated, thereby breaking the weak hydroxyl bond. The hydroxyl bond reacted with the methylene group and generated carboxyl groups. The number of oxygen-containing functional groups such as C=O increased, enhancing the hydrophilicity of the coal samples. These findings indicated that low-temperature oxidation increased the hydrophilicity of coal samples and decreased the hydrophobicity, ultimately decreasing the floatability of low-rank coals. As low-temperature oxidation times increased, the number of hydrophilic functional groups on the surface of coal samples increased and the hydrophobicity decreased, which eventually decreased the floatability of low-rank coals.

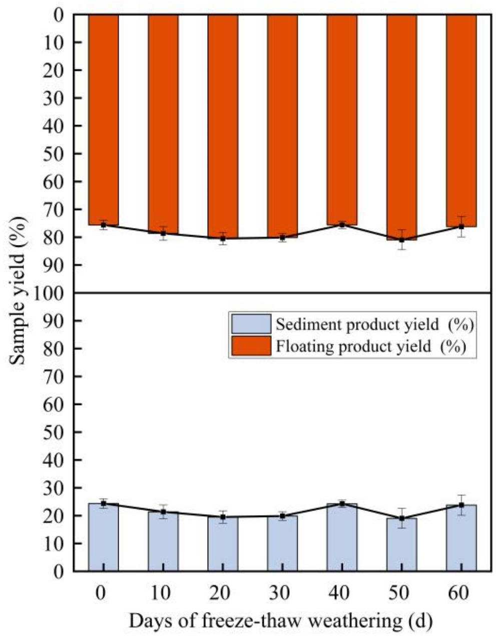

Fig. 14. Trend of coal sample yield at different freeze-thaw weathering times

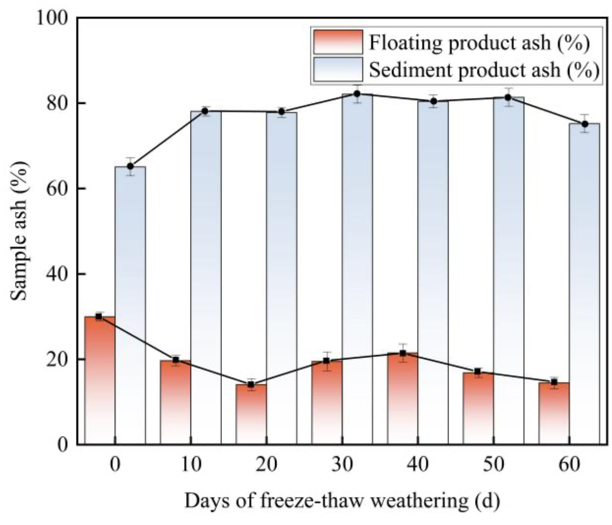

Fig. 15. Trend of ash content of integrated gangue products with different freeze-thaw weathering times

3.3.2. SEM

Fig. 17 shows the SEM results of coal samples with different low-temperature oxidation times. Due to low-temperature oxidation, the low-rank coal particles dissociated. The surface of coal samples was dense and smooth in the absence of low-temperature oxidation. As the time increased, the surface fractured and damaged, forming fissures, gullies, voids and 'bubble-like' pits. The gas generated from the cracking of coal samples fructured (Jun et al., 2019), exposing internal elements (Shen et al., 2020).

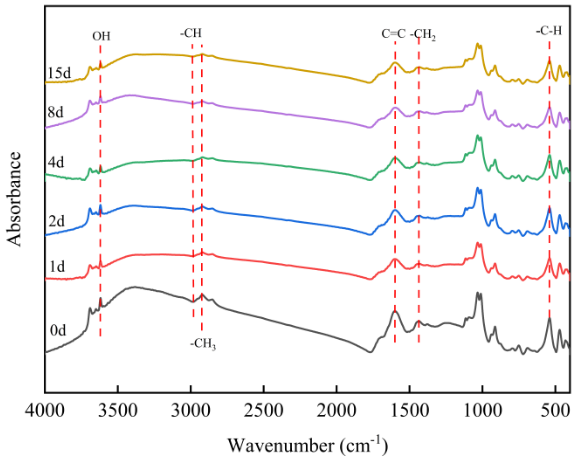

Fig. 16. FTIR of coal samples with different low-temperature oxidation times

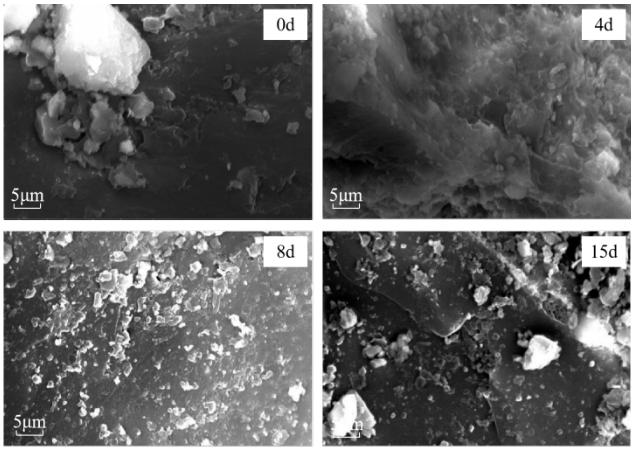

Fig. 17. Scanning electron microscopy (SEM 20,000x) of coal samples with different low-temperature oxidation times

3.3.3. Zeta potential tests

Fig. 18 shows that absolute zeta potential became more negative from −93.60 (mV) to −106.06 (mV) as the low-temperature oxidation times increased from 4 days to 8 days. These values tended to stabilise gradually after 8 days. Moreover, the increasing repulsive force between microscopic particles, the transition of slime from the particle surface to the slurry phase, and the relative decrease in the distribution of coal particles resulted in poor flotation recoveries of low-rank coals (Zheng et al., 2020).

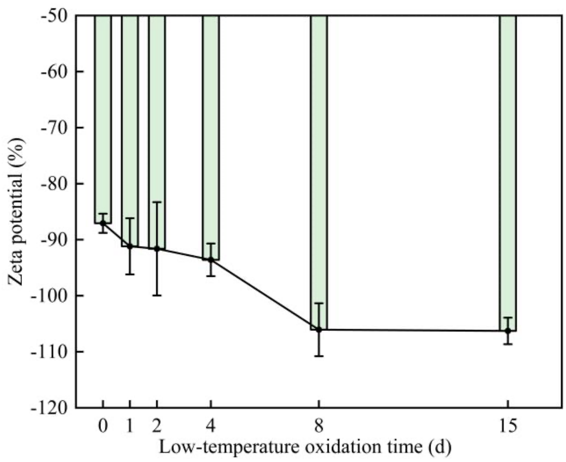

Fig. 18. Zeta potential variation of coal samples with different low-temperature oxidation times

3.3.4. Contact angle measurements

The contact angle of coal samples without low-temperature oxidation was the largest at approximately 52°, which decreased to approximately 48° after 15 days. As low-temperature oxidation continued, the contact angle on the surface of coal samples gradually decreased (Fig. 19). A decrease in contact angle indicates a decrease in the hydrophobicity of the particle, which results in lower flotation yield. These ultimately reduced the flotation yield of low-rank coal.

3.3.5. Laboratory flotation tests

The flotation yield of low-rank coal was approximately 25%, which decreased to approximately 15% after 15 days. The ash content in fresh low-rank coal was approximately 20%, which increased to approximately 25% after 15 days. These findings indicated that the quality of coal-series kaolin considerably changed as the low-temperature oxidation time increased. The flotation yield decreased obviously (Fig. 20), and the trend was most obvious after 0 and 1 day; the yield stabilised after 4 days. The ash content in the coal particles gradually increased, as shown in Fig. 21.

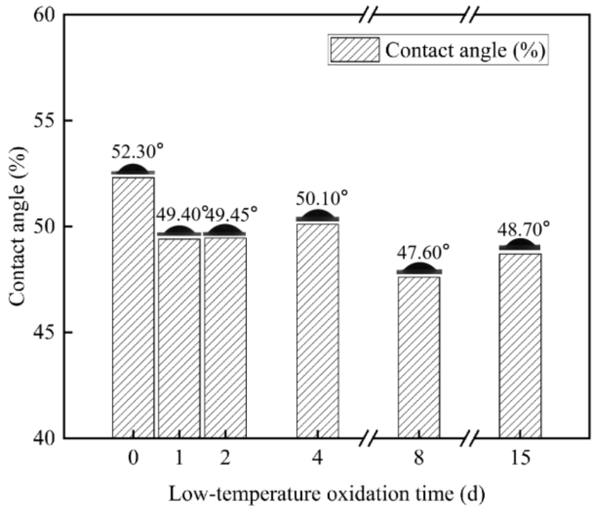

Fig. 19. Contact angle variation of coal samples with different low-temperature oxidation times

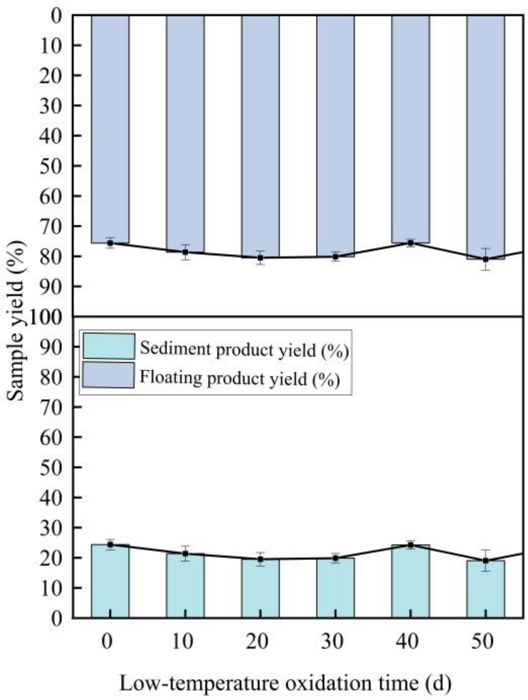

Fig. 20. Variation of yield of coal samples with different low-temperature oxidation times

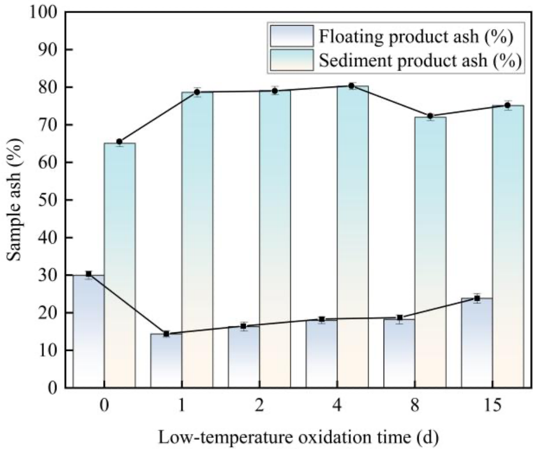

Fig. 21. Variation of ash content of coal samples with different low-temperature oxidation times

4. Mechanistic analysis of weathering process

Unlike short-term freeze–thaw weathering, after short-term natural weathering and low-temperature oxidation, the coal surface appeared cracked. The internal elements were exposed, and the hydrophobic $-CH_3$, $-CH_2$ and -C-H functional groups gradually reduced, mainly during natural weathering from 4 to 6 months and low-temperature oxidation from 4 days to 8 days. As weathering continued, a small amount of carbon monoxide and carbon dioxide gas overflowed, generating a small amount of heat (100℃–200℃). The amount of carbon monoxide and carbon dioxide gas increased exponentially, releasing large amounts of heat (200℃–350℃) (Xianliang et al., 2023, Cai et al., 2019a). As shown in Fig 22, the low-rank coal weathered considerably during oxidation, during which -OH in the coal reacted with oxygen to generate C=O. During this time, the -OH content sharply decreased and C=O reacted with oxygen to eventually generate -COOH (Zhou et al., 2017). The free OH combined with the hydroxyl group to generate water and decreased the hydrogen content, thereby increasing the surface hydrophilicity.

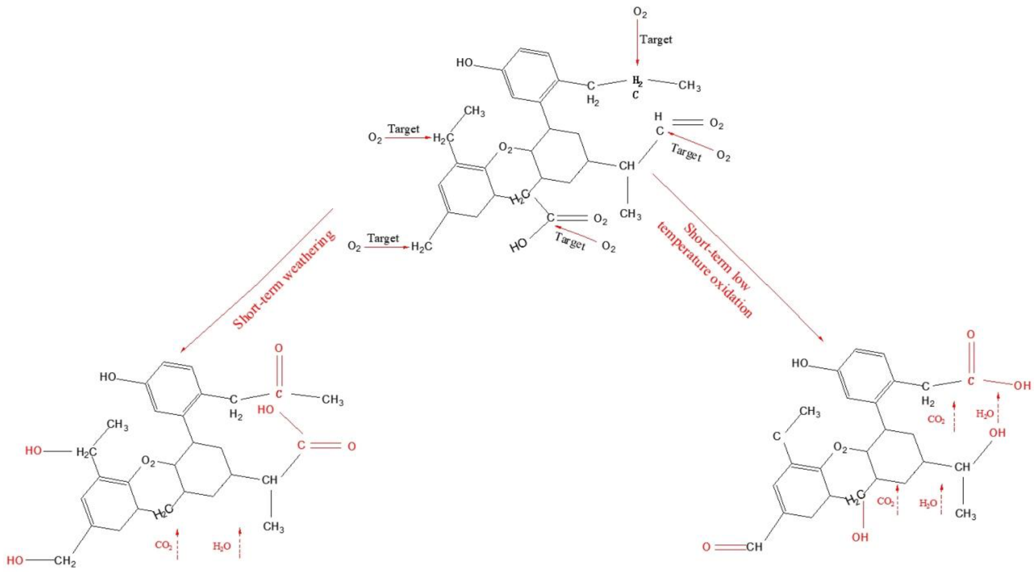

Fig. 22. Schematic diagram of weathering mechanism

5. Conclusions

Coal-series kaolin is an important nonmetallic mineral resource. Herein, the effects of common types of weathering—short-term natural weathering, low-temperature oxidation and freeze–thaw weathering—on the effectiveness of the flotation decarburization of coal-series kaolin were investigated. Based on the above-mentioned experimental results, the following conclusions are drawn:

1. Natural weathering considerably impacted the recovery of low-rank coals from coal-based kaolin. As the natural weathering time increased, the ash content in coal sinks after flotation increased by approximately 10%, decreasing the coal floatation yield by 17.66%. The floatation yield increased to 93.30%, which is a decrease in the yield and deterioration of the quality. SEM, zeta potential and FTIR spectroscopy revealed that the natural weathering changed the microscopic appearance of the coal particle surface. Moreover, the size of the coal particles dispersed in water decreased and gradually stabilised. This decreased the surface hydrophobicity and deteriorated the floatability of low-rank coal.
2. Freeze–thaw weathering only slightly impacted the recovery of low-rank coal from coal-series kaolin. SEM, zeta potential and FTIR spectroscopy confirmed the surface and size of the coal particles did not change considerably. The system stability also changed slightly, which did not affect the floatability.
3. Low-temperature oxidation considerably impacts the recovery of low-rank coal. As the oxidation time increased, the flotation yield and floatability gradually decreased. Zeta potential, SEM and FTIR spectroscopy revealed that continuous low-temperature oxidation destroyed the surface of coal samples and increased the number of hydrophilic functional groups. Consequently, the hydrophobicity decreased and the flotation deteriorated, thereby affecting the flotation yield of the coal-series kaolinite in recovering the low-rank coal.

The next phase of this study will focus on developing control strategies to mitigate the effects of weathering on flotation efficiency. Some control strategies include grinding, ultrasonic surface cleaning and removal of oxidation layers. Current experiments were conducted under laboratory-simulated conditions. Meanwhile, future work will involve collecting field samples from typical coal mining areas; the samples from varying depths and being subjected to weathering for different durations will be collected. This study aims to gain a better understanding of the floatability of weathered coal and to support lifetime prediction models for the flotation performance of such coal.

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