Structure and Chemical Composition of Low-silicon White Mud Based on Technology of Black Liquor Combustion Desilication

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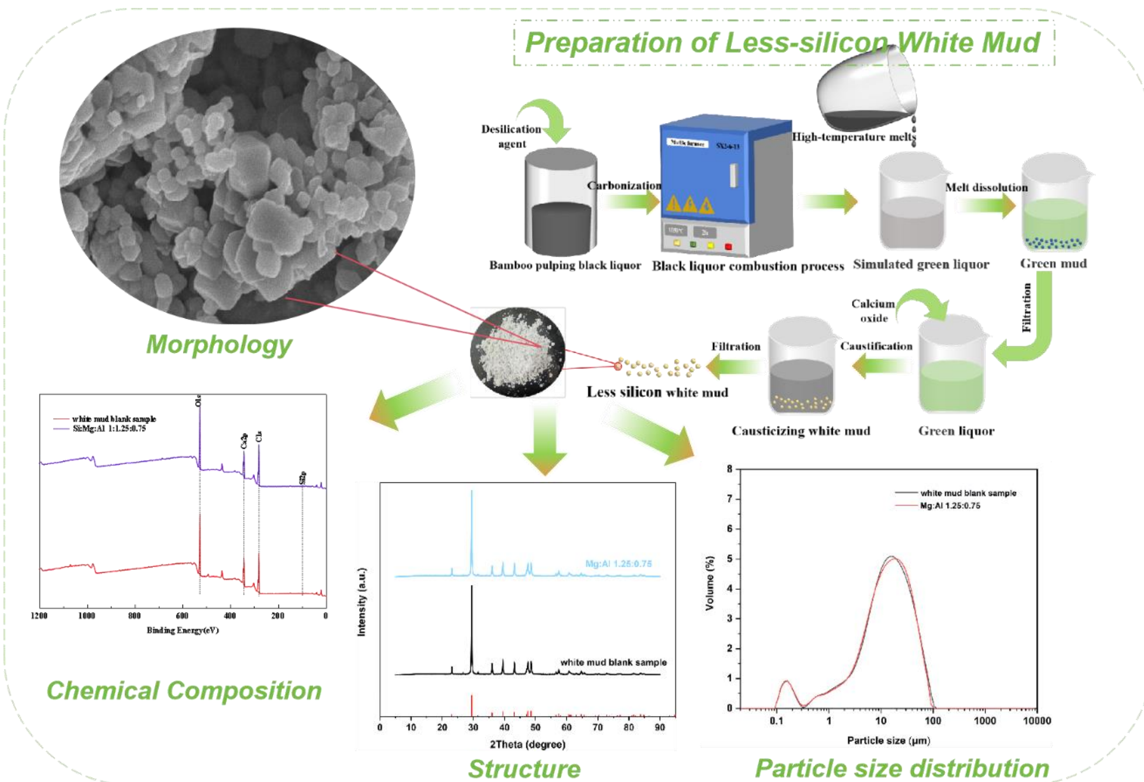
GRAPHICAL ABSTRACT



Structure and Chemical Composition of Low-silicon White Mud Based on Technology of Black Liquor Combustion Desilication

Yanghua Pei,[a] Yongjian Xu,[a,b,c,d,]* Ying Zhang,[a] YangXu,[a] Hao Chen,[a] and Xin Chen [a]

The sulfate process for bamboo pulp production encounters silicon interference during the pulping stage, necessitating desilication before the alkali recovery section. This study investigated the desilication of bamboo pulping black liquor (BPBL), demonstrating that silicon was removed effectively by adding Mg/Al compound desilication agent to the BPBL *via* burning at high temperature. The desilication extent can reach 84% when the desilication agent is added in the proportion of Mg/Al (magnesium sulfate and sodium metaluminate) in the ratio 1.25:0.75. Subsequently, less-silicon white mud (LSWM) can be prepared after black liquor (BL) combustion desilication. LSWM was compared with normal WM as a blank sample, and the morphology and physicochemical properties of two kinds of WM were characterized. X-ray diffractometry revealed that both types of WM are primarily composed of calcite crystalline $CaCO_3$ and hydrated $CaSiO_3$. Scanning electron microscopy, X-ray photoelectron spectroscopy, and particle size analysis demonstrated the reduced content of $CaSiO_3$ generated in the LSWM. This result indicated that the Mg/Al compound desilication agent removed the silicon from the green liquor (GL), thereby reducing the residual silicon in WM. This reduction is beneficial to the calcination recovery and comprehensive utilization of WM.

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Keywords: Black liquor combustion desilication; Less-silicon white mud; Structure; Chemical composition; Desilication effect

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INTRODUCTION

According to the report of China Paper Association, the total volume of pulp production was 85.87 million tons in 2022, with an increase of 5.01% over the previous year (Wang *et al*. 2015). However, the pulping industry faces a significant challenge in the form of white mud (WM), a by-product of the alkali recovery system. Approximately 0.47 tons WM are generated when 1 ton pulp are obtained. A mass of WM, more than 3 million tons in 2021 (Zhao 2016), was produced in the non-wood pulping industry.

During the running of an alkali recovery system, concentrated black liquor (BL), which consists of organic and inorganic salts, is pumped into the alkali recovery boiler to recover the inorganic salts *via* atomizing, drying, burning, oxidizing, and removing organic matter (Cha *et al*. 2018). The recovered molten inorganic salts are incorporated with water to form a clear and transparent liquid with a color of green, which is defined as green liquor (GL) and mainly consists of Na_2CO_3. The GL is treated by lime (CaO) to initiate the causticizing reaction. Subsequently, the white precipitate from causticizing reaction, mainly consisting of $CaCO_3$, is removed to recover the supernatant from causticizing reaction (Ke *et al*. 2009), which is defined as white liquor and can be reutilized in cooking process (Fig. 1).

In addition to $CaCO_3$, WM contains excess lime, calcium silicate, residual sodium hydroxide, and a small amount of sodium sulfide, aluminum, iron, magnesium compounds, and dust impurity from the causticization process (Ai *et al*. 2003). The calcium silicate will decrease the quick lime activity when the WM is calcined to produce quick lime, which will affect the calcination and reuse of WM. Among them, calcium silicate, which is produced in the GL causticizing process from non-wood pulping and contained in WM, will affect the washing and filtration process of WM, resulting in a high content of alkali containing in the WM. In addition, the moisture content of WM cannot achieve a satisfactory value following filtration due to the present of $CaSiO_3$. As a result, the calcination, recycling, and comprehensive utilization of WM are affected (Wang *et al*. 2022). Hence, many pulp mills have to landfill WM. Unfortunately, the landfilling of WM will occupy a large amount of land. What is more, the alkaline and corrosive nature of landfilled WM will lead to the alkaline contamination of the land, which will endanger the environmental safety, due to the strongly alkaline and corrosive of WM (Zhu *et al*. 2015). Because of the difference of silica content in wood and non-wood lignocellulosic raw materials, above-mentioned “silicon interference” is absent in the WM derived from wood pulping, nonetheless, is present in the WM derived from non-wood pulping.

White mud (WM) can be better resourced if there is no silicon interference. There is a lot of research on the application of WM. It can be reprocessed as raw material or auxiliary material in other fields, such as cement production (Fu *et al*. 2003), construction ceramics production (Sun *et al.* 2012), page brick production (Zhang *et al.* 2011), as well as refined WM used as filler for papermaking (Yang *et al.* 2012), plastic additives (Lin *et al.* 2010), and flue gas desulfurization (Ren *et al.* 2009). If the latter WM is to be converted into a usable resource, the development of desilication technology must be improved to decrease the silicon content in the WM (Cha *et al.* 2018).

Therefore, it is essential to identify an appropriate approach to eliminate $CaSiO_3$ from WM, thereby enhancing the reutilization efficiency of WM. Many desilication methods have been proposed, such as CO_2 method (Xia *et al.* 2012; Li *et al.* 2014), pre-causticization method (Xia *et al.* 2011; Xu *et al.* 2013), and Mg/Al compound desilication method (Lin *et al.* 2014; Xu *et al.* 2013). Aluminum sulfate ($Al_2(SO_4)_3$) and sodium aluminate ($NaAlO_2$) have been used as aluminum salt desilication agents in BL from wheat straw pulping to remove the silicon via BL combustion process. The desilication ratio in the process is 53.02% (Xu *et al.* 2015). Magnesium sulfate ($MgSO_4$) and oxide magnesium (MgO) were used as magnesium salt desilication agents to desilicate *via* the BL combustion process. The former was better than the latter, and the desilication ratio could reach 82%. In addition, self-causticization and desilication of GL were realized with $MgSO_4$ as the desilication agent and sodium borate ($Na_2B_4O_7 \cdot 10H_2O$) as the self-causticization agent (Xu *et al.* 2015; Xu *et al.* 2016). The combination of sodium aluminate and basic magnesium carbonate can be used to remove the silicon, and exhibiting an excellent desilication effect. Silica was removed up to 84 wt% with a ratio of Mg/Al as 5:3 in desilication agent and the dosage of desilication agent was 1.004 wt% of the solid content in BL. In addition, the desilication agent is an important residual in GL (Xu *et al.* 2022).

Moreover, BL combustion desilication is more suitable to remove silicon than direct desilication in GL. A large proportion of silicon can be removal *via* BL combustion desilication processing residual silicon, even with a low content, will affect the calcination and further utilization of WM. Existing methods of BL desiliconization are faced with issues such as the extremely low concentration of sodium silicate involved in the reaction, high consumption of chemicals, high costs, and the impact on the pH value of the GL. These issues result in a high content of $CaSiO_3$ in the WM, leading to poor water filtration, difficulty in washing, high residual alkali, high energy consumption during calcination, and low active calcium oxide content in the calcined quicklime, thus reducing its usability (Xia *et al.* 2014; Fang *et al.* 2016). Hence, it is necessary to further carry out GL desilication to improve the reuse of WM.

This study proposes a method for silicon removal from GL to minimize the silicon content in bamboo pulping WM, and obtain the desilicated WM (*i.e.*, less-silicon white mud, LSWM). The morphology, particle size distribution, elemental composition and content, and phase structure of the LSWM were analyzed through SEM-EDS, XRD, XPS, and particle size analysis. The purpose of this study is to analyze the impact of $CaSiO_3$ content in WM on the particle size and properties of $CaCO_3$ in WM, comparing the conventional GL desiliconization process and BL combustion via GL desilication process. By analyzing the influence of $CaSiO_3$ content corresponding to the desiliconization ratio on the structure, particle size, and water filtration of $CaCO_3$ in non-wood pulp WM, the correlation between structure and performance is established. The effects of mixed $CaSiO_3$ on WM are elucidated, and the feasibility of the subsequent WM causticizing process after BL combustion via GL desilication process is verified. This demonstrates the resource utilization value of non-wood pulp WM based on the BL combustion via GL desilication process.

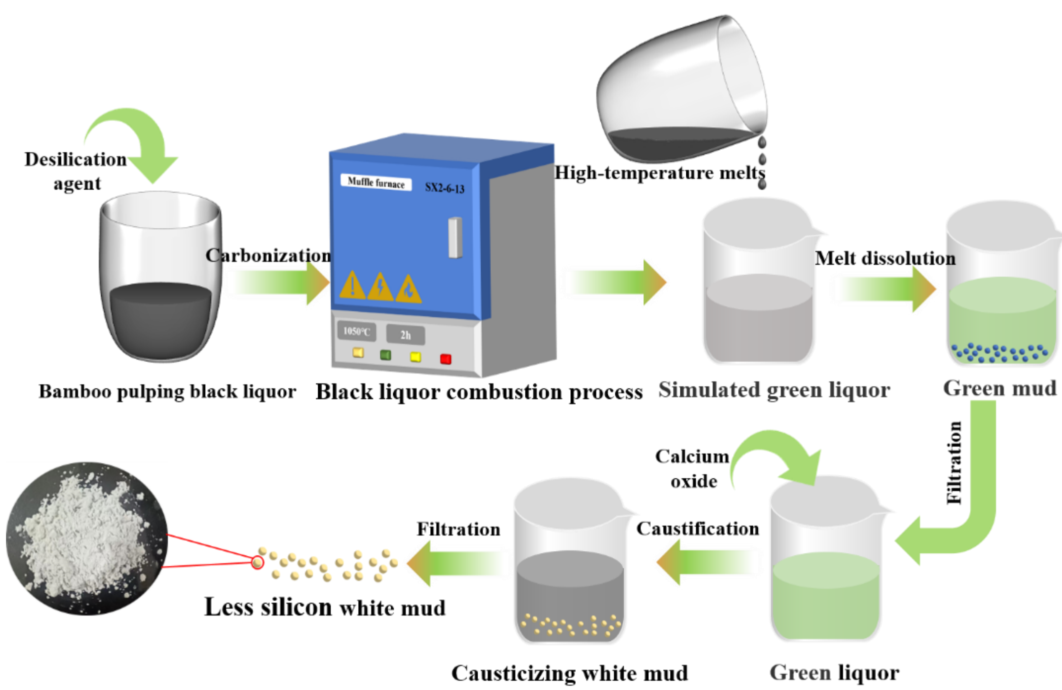


Fig. 1. Preparation of less-silicon white mud from green liquor (GL) desilication *via* black liquor (BL) combustion process

EXPERIMENTAL

Materials

The bamboo pulping BL had a solids content of 41.5 wt%, with the organic content accounting for 68.77% and the inorganic content for 31.23%. Among the inorganic content, the silicon content of 0.33% (calculated as SO_2), was sourced from the high-temperature degradation section of Guizhou Chitianhua Paper Industry Company (China). In this section, the macromolecules such as cellulose and lignin in BL were degraded under high temperature. Basic magnesium carbonate ($4MgCO_3{\cdot}Mg(OH)_2{\cdot}5H_2O$) and sodium aluminate ($NaAlO_2$) were obtained from Sinopharm Chemical Reagent Co. Ltd. (Shanghai, China). Sodium carbonate (Na_2CO_3) was purchased from Tianjin Damao Chemical Reagent Factory (Tianjin, China). Simulated GL was prepared by dissolving sodium carbonate in water to achieve a concentration of 110 g/L. Calcium oxide (CaO), nitric acid (HNO_3), and hydrogen peroxide (H_2O_2) were procured from Cologne Chemicals Company (Chengdu, China).

Preparation of Clear Green Liquor

The desilication effect of different proportions of desilication agents is shown in Table 1. The optimal proportion was determined based on the silicon content after desilication and the content of residual desilication agent in the GL. The results showed that the desilication ratio could reach 84% with a ratio of Mg/Al as 1.25:0.75 in the compound desilication agent. Under these conditions, the content of residual desilication agent in the GL was also relatively low.

Table 1. Desilication Ratio of the Mg/Al Compound Desilication Agent

Scheme	Si (expressed as SiO_2/mol)	Mg (expressed as SiO_2/mol)	Al (expressed as SiO_2/mol)	Desilication ratio (%)
1	1	1.25	0.5	82%
2	1	1.25	0.75	84%
3	1	1.25	1	88%
4	1	1.25	1.25	87%
5	1	1.25	1.5	90%

Preparation of Less-silicon White Mud

The clear GL obtained by filtration was used for causticization. Then CaO was added to the GL with a ratio of 105 wt.% based on GL weight and stirred vigorously using a glass rod. Then, the mixture was put into the oil bath to complete the causticization reaction and maintained at 100 °C for 2 h. After causticization and depositing for 1 h, the WM was washed and filtered using deionized water to obtain LSWM with minimal residual alkali content. Finally, the LSWM was dried in a fan dryer and ground in a ball mill for 30 min.

Morphology and Structural Characterization of Less-silicon White Mud

The scanning electron microscopy (SEM) (SU8100, Hitachi, Tokyo, Japan) was used for observing the surface microscopic morphology of LSWM, and energy-dispersive X-ray spectroscopy (EDS) was applied to qualitatively analyze the elements in the surface micro-area of LSWM before and after desilication.

The X-ray photoelectron spectrometer (XPS) (AXIS Supra, Kratos, England) was carried out to characterize the content of C, O, Ca, Si, and other elements in the WM before and after desilication.

The X-ray diffractometer (XRD) (Ds Advance, Bruker, Germany) was conducted to investigate the chemical composition and structure of WM. The XRD patterns were recorded with a scanning speed of 0.02°/s over a 2θ range of 5 to 90°.

Particle Size Analysis

The laser particle size analyzer (Mr2000, Mastersizer, UK) was used to detect the particle size distribution of the WM. The instrument parameters were set using a refractive index of 1.69, which corresponds to the refractive index of $CaCO_3$, the main component of WM.

RESULTS AND DISCUSSION

Morphology of Less-silicon White Mud

SEM images of the WM blank sample and LSWM are shown in Fig. 2. CaO generates $Ca(OH)_2$ and OH- when it reacts with water. Subsequently, the reaction between $Ca(OH)_2$ and the simulated GL, which mainly contains Na_2CO_3, produces $CaCO_3$ precipitate, known as WM. The results show that the microscopic morphologies of WM before and after desilication are quite similar; both exhibit granular precipitates with sizes in the nanometer range. The square granular form of particles precipitates, which is particularly evident in Fig. 2a. Presumably, the image shows $CaCO_3$ particles produced during the causticization of GL. Notably, numerous square granular precipitates can be observed in both Fig. 2a and Fig. 2b, displaying similar apparent morphologies before and after desilication.

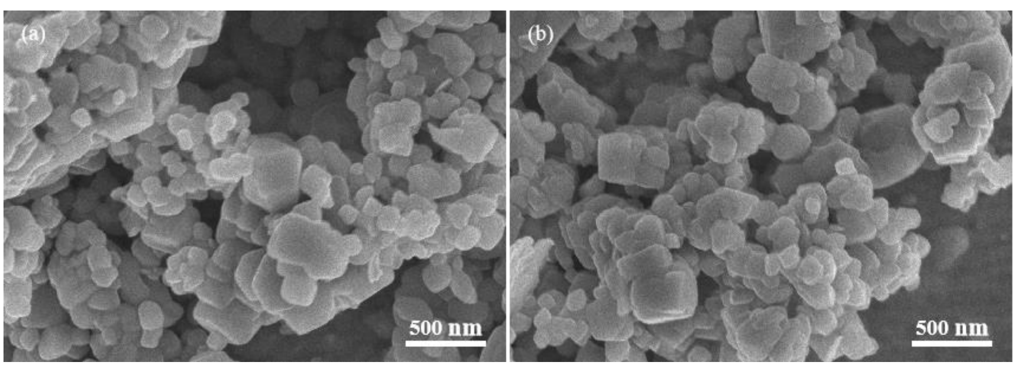
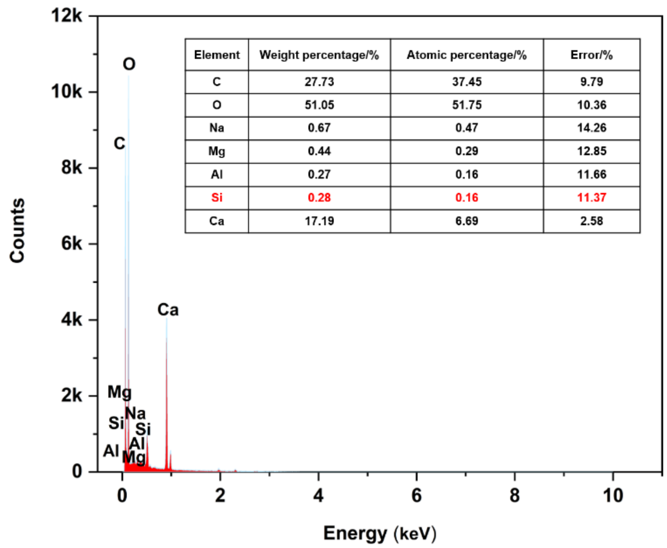


Fig. 2. SEM images of white mud: (a) white mud blank sample; (b) less-silicon white mud prepared with a ratio of Mg/Al as 1.25:0.75 in compound desilication agent

Chemical Composition and Structure of White Mud

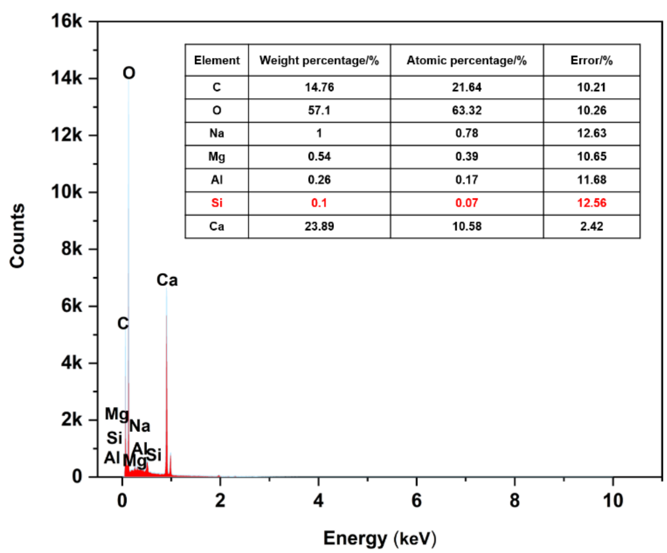
Chemical composition and distribution of silicon elements

EDS analysis was carried out to analyze and detect the elements present in WM samples, aiming to evaluate the changes in WM elements composition after desilication. The results are shown in Fig. 3, 4, and 5. As observed in Fig. 3, the WM blank sample mainly contained C, O, Ca, Si, Al, Mg, Na, and a few other elements, with C, O, and Ca accounting for a large proportion due to the presence of $CaCO_3$ in WM. By calculating and analyzing the EDS data, it has been found that in the composition of the blank WM, the ratio of $CaSiO_3$ to $CaCO_3$ was 1:40.8, whereas in the LSWM, the ratio was 1:150.1. The silicon content in the LSWM is much lower than in the blank WM. The elemental composition of LSWM was similar to that of WM blank sample; however, there were some differences in the element contents, in particular the Si content in LSWM is relatively reduced compared to the WM blank sample (Fig. 4). This reduction can be attributed to the decrease in $CaSiO_3$ content in the LSWM, which is a result of GL desilication via the BL combustion process.



Element	Weight percentage/%	Atomic percentage/%	Error/%
C	27.73	37.45	9.79
O	51.05	51.75	10.36
Na	0.67	0.47	14.26
Mg	0.44	0.29	12.85
Al	0.27	0.16	11.66
Si	0.28	0.16	11.37
Ca	17.19	6.69	2.58

Fig. 3. EDS analysis of white mud blank sample



Element	Weight percentage/%	Atomic percentage/%	Error/%
C	14.76	21.64	10.21
O	57.1	63.32	10.26
Na	1	0.78	12.63
Mg	0.54	0.39	10.65
Al	0.26	0.17	11.68
Si	0.1	0.07	12.56
Ca	23.89	10.58	2.42

Fig. 4. EDS analysis of less-silicon white mud prepared with a ratio of Mg/Al as 1.25:0.75 in compound desilication agent

SEM-EDS surface scanning was used to determine the elemental distribution of Si in LSWM. As shown in Fig. 5, the element Si was widely distributed in the edge area of LSWM particles, suggesting that the generated $CaSiO_3$ may be present on the surface of WM particles in the form of adsorption.

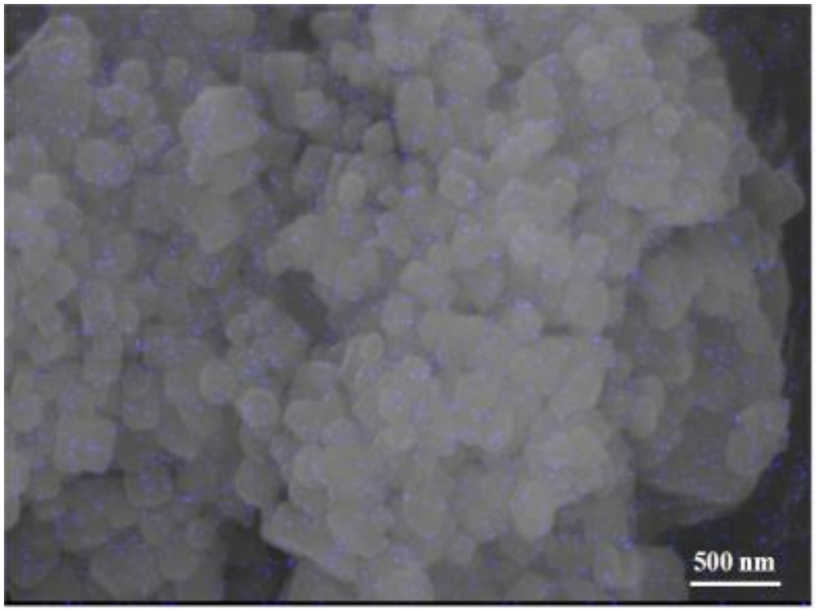


Fig. 5. Si element distribution map of less-silicon white mud prepared with a ratio of Mg/Al as 1.25:0.75 in compound desilication agent

XPS analysis of white mud

To analyze the effect of $CaSiO_3$ content on the particle size and performance of $CaCO_3$ particles in WM, XPS was used to characterize the presence and chemical state of the main elements in the WM and the changes after desilication. The XPS spectrum of WM blank sample is shown in Fig. 6. The valence states of C1s, O1s, Ca2p, and Si2p elements in WM blank sample can be determined from the spectrum, with each element exhibiting a large peak area and varying peak intensities. Based on the above analysis, it can be inferred that the WM blank sample was mainly composed of $CaCO_3$ and an unavoidable amount of $CaSiO_3$, which includes C, O, Si, Ca, and other elements. This composition results in the inability to calcine and recycle the WM directly. Therefore, it is necessary to reduce or completely remove the silicide in the WM to enable its effective utilization.

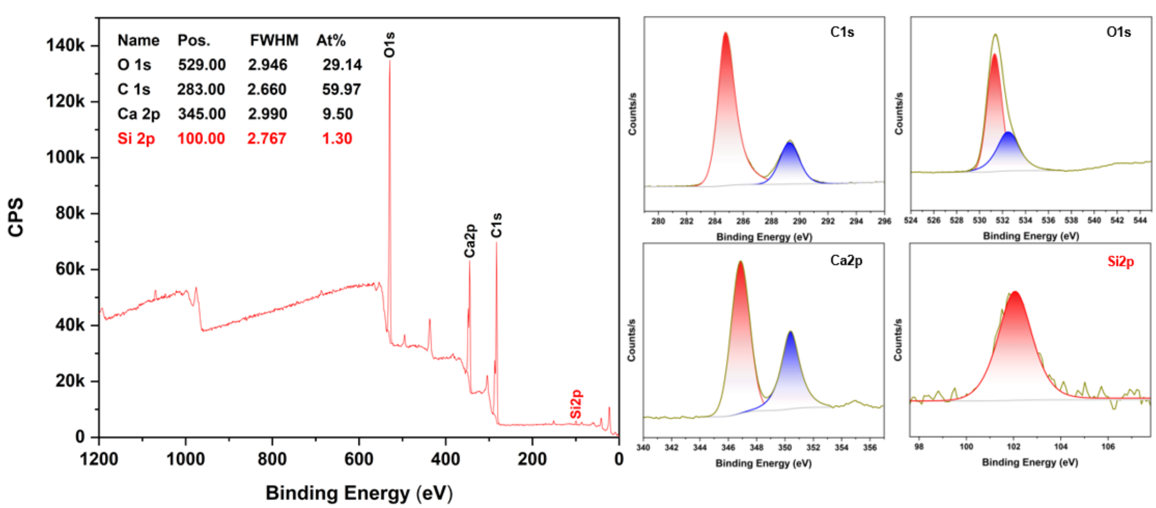


Fig. 6. XPS spectrum of white mud blank sample

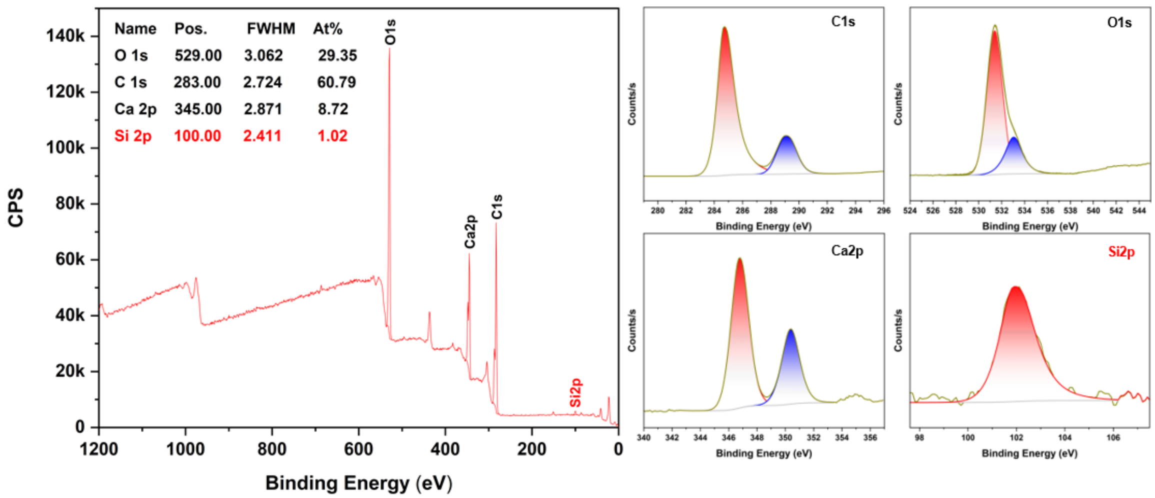


Fig. 7. XPS spectrum of less-silicon white mud

Previous studies have shown that a good desilication effect can be obtained through GL desilication *via* the BL combustion process (Xu *et al.* 2022), and the LSWM is subsequently obtained through the causticization of less-silicon clarified GL. The XPS spectrum of LSWM, prepared with a ratio of Mg/Al as 1.25:0.75 in the compound desilication agent, is shown in Fig. 7. There was no difference between the LSWM and WM blank sample in terms of element composition and chemical state. Both of them contained C1s, O1s, Ca2p, and Si2p elements, and the peak area of C1s, O1s, Ca2p in the LSWM was not significantly different from those in the WM blank sample. However, the peak area of the Si2p element in LSWM was reduced, indicating a decrease in the content of Si element. This decrease suggests that the content of $CaSiO_3$ in the LSWM was lower after desilication. The XPS results of both WM blank sample and LSWM demonstrate that the desilication process had a good desilication effect, which provides new ideas and solutions for the recycling of WM.

Structure of white mud

The chemical compositions of WM blank sample and LSWM were analyzed using SEM-EDS and XPS, showing that the compound desilication agent had a good effect on the desilication experiment. However, there was no specific detection and analysis of the crystal structures of two types of WM. Therefore, XRD was used to characterize the crystal forms of $CaCO_3$ and $CaSiO_3$ in the WM.

The XRD patterns of WM black sample and LSWM, prepared with a ratio of Mg/Al as 1.25:0.75 in compound desilication agent, are shown in Fig. 8. The crystal forms of both WM had not changed significantly. The peak types were relatively orderly with fewer heterogeneous peaks, and the peak positions and characteristic peaks were clear. One of the highest characteristic peaks appeared with high peak intensity at 29°, approaching 30° in 2θ for both WM, the characteristic peaks of both WM basically conform to the peak location of $CaCO_3$ standard card (JCPDS card No. 47-1743). This demonstrates that the $CaCO_3$ generated in the two types of WM was in the calcite crystal form. Xu *et al.* (2022) showed that $CaSiO_3$ has a strong diffraction peak at 29.3° in 2θ, which overlaps with the peak positions of $CaCO_3$ in both WM specimens. Therefore, combining the results of elemental analysis, it can be concluded that hydrated calcium silicate existed in both WM. In addition, the other peak positions were basically the same as those of hydrated calcium carbonate. Unfortunately, the differences between peaks of the two WM cannot be discerned from the XRD patterns, and it is speculated that there are the following possible reasons for this observation.

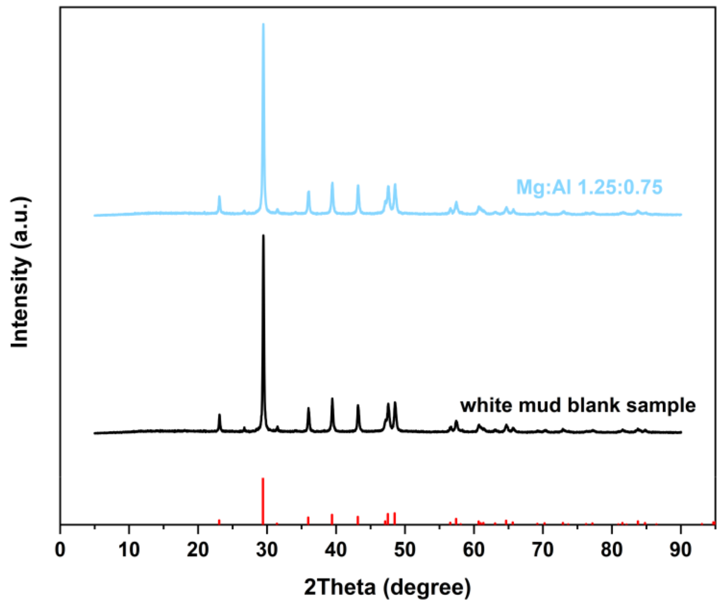


Fig. 8. XRD patterns of white mud samples

Both types of WM samples exhibited calcite crystal forms, with their composition primarily consisting of crystalline $CaCO_3$. However, the $CaCO_3$ is invariably intermixed with trace amounts of $CaSiO_3$, present as a solid solution rather than forming a distinct crystal structure within the WM. Secondly, the diffraction peak positions of hydrated calcium silicate and calcite overlap significantly, indicating that their crystal forms are closely aligned. Comprehensive testing and analysis of WM samples lead to the conclusion that the primary constituents of WM are crystalline calcite and hydrated calcium silicate.

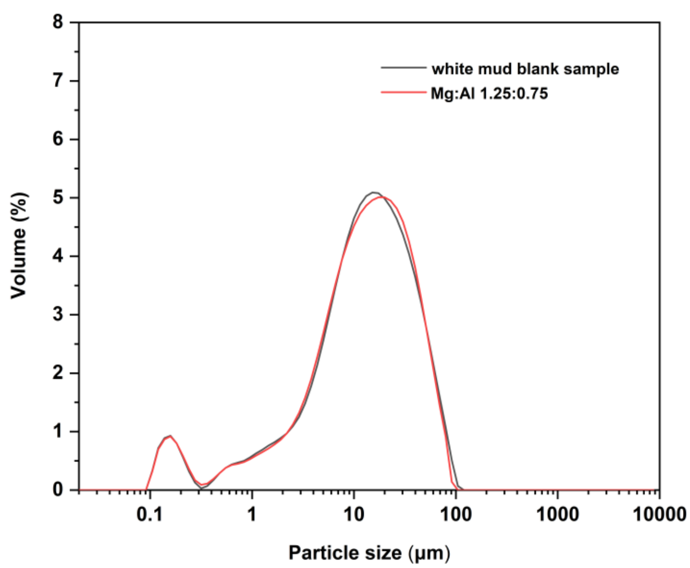


Fig. 9. Particle size distribution of white mud samples

Particle Size Analysis of White Mud

The molecular and morphological characteristics of WM particles, including surface morphology, average particle size, and particle size distribution, can be observed and analyzed. Figure 9 presents the results of the WM blank sample and the LSWM prepared with a ratio of Mg/Al as 1.25:0.75 in the compound desilication agent.

The particle size distribution of both WM samples ranged primarily from 10 to 100 μm, with the particle sizes of both WM being nearly identical. The particle size curves exhibited a generally normal distribution, with a prominent peak appearing in the overall distribution map. The particle size curves of the WM blank sample and the LSWM almost overlapped. As shown in Fig. 9, the particle size curve range spanned from 10 to 40 μm, with the particle size curve of the LSWM slightly shifted to the right compared to that of the WM blank sample. The average particle size of the WM blank sample was 14.27 μm, while the average particle size of the LSWM, prepared after desilication, was 14.32 μm. The slightly larger average particle size of LSWM compared to WM blank sample may be attributed to the adsorption of desilication agent on the surface of WM particles after the desilication process.

CONCLUSIONS

1. Based on scanning electron microscopy – energy-dispersive X-ray spectrometry (SEM-EDS) energy spectrum analysis, surface sweep results, and the particle size distribution of white mud (WM), it is hypothesized that the particle size of low-silicon white mud (LSWM) exhibited decreases in the range of 10 to 20 μm. This reduction may be attributed to the small size of $CaSiO_3$ particles. After desilication, the Si element content in GL was reduced, leading to a decrease in the content of $CaSiO_3$ generated in the LSWM. Consequently, it is speculated that the reduced particles may be precipitated $CaSiO_3$ or $CaSiO_3$ adsorbed on the surface of $CaCO_3$. These $CaSiO_3$ particles are likely washed off during the washing process, resulting in an increase in WM particle size after desilication.
2. The X-ray diffraction (XRD) characterization of the WM blank sample and LSWM showed that both were primarily composed of $CaCO_3$ in calcite crystal form and hydrated $CaSiO_3$. The characterization results from SEM, XPS, and particle size analysis confirmed that the content of $CaSiO_3$ generated in LSWM was reduced, indicating that the use of the Mg/Al compound desilication agent can effectively remove silicon from both green liquor (GL) and WM, which is beneficial for calcination recovery and comprehensive utilization of WM.

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