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Comparative Study on Different Ammonia Removal Technologies for Electrolytic Manganese Residue

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Wet process ammonia removal and pyrometallurgical ammonia removal are two mainstream technologies for ammonia nitrogen treatment of electrolytic manganese residue (EMR). However, systematic comparative studies based on identical raw material conditions are currently insufficient, which cannot provide adequate support for enterprises in process selection. Taking EMR generated by an enterprise in Yichang as the research material, experiments on wet process and pyrometallurgical ammonia removal were carried out. The ammonia nitrogen removal performances of the two processes were compared and analyzed, and a multi-dimensional comprehensive evaluation was conducted in combination with process characteristics, resource utilization potential, and disposal cost. The results show that under the optimized conditions of calcium carbide slag dosage of 10 %, stirring time of 30 min, stirring speed of 300 r/min, and liquid-solid ratio of 20, the nitrogen removal rate of the wet process reaches 86.35 %. For the pyrometallurgical process, the nitrogen removal rate is 93.53 % under the calcination condition of 500 ℃ for 90 min. The wet process suffers from the generation of high ammonia nitrogen waste liquid, large reagent consumption, and limited resource recovery pathways for the treated residue. By contrast, the pyrometallurgical process can recover NH_3 from flue gas to prepare ammonia water, and the calcined slag possesses broad prospects for resource utilization, whereas it has the drawback of high energy consumption. Overall, pyrometallurgical ammonia removal technology is more suitable for engineering scenarios with large-scale treatment capacity and high requirements for residue resource utilization. The wet process still has applicability for scenarios featuring small-scale treatment and limited investment. This study provides a multi-dimensional decision-making reference for the scientific selection of ammonia removal processes for EMR.

Keywords: electrolytic manganese residue; ammonia nitrogen; ammonia removal; wet-process; pyrometallurgical-process.

1. INTRODUCTION

Electrolytic manganese residue (EMR) is a waste residue generated during the production of electrolytic manganese. According to statistics, the cumulative stockpile of EMR in China has reached 160 million metric tons, with nearly 10 million metric tons added annually [1]; however, the comprehensive utilization rate remains below 10 % [2]. EMR contains excessive levels of ammonia nitrogen, has a high moisture content, and its soluble components are prone to migration [3 – 5]. Long-term open-air stockpiling poses a serious threat to human health, ecological stability, and the surrounding environment [6 – 8]. Therefore, achieving efficient removal of ammonia nitrogen from EMR is a critical issue that electrolytic manganese enterprises must address to achieve green and sustainable development.

Currently, the industry has not yet established an ammonia removal process that can both reliably ensure compliance with ammonia-nitrogen discharge standards and be widely adopted [9]; however, wet ammonia removal and pyrometallurgical ammonia removal are the two relatively mainstream technologies. Wet ammonia removal involves using water washing to transfer ammonia nitrogen into the liquid phase; after solid-liquid separation, the ammonia nitrogen content in the slag is reduced. Alternatively, water washing combined with alkaline agents can convert ammonia nitrogen into NH_3, which then volatilizes, thereby achieving ammonia removal. Wang Jiawei [10] studied the ammonia removal effect of pure water washing on EMR. Kang Zhipeng et al. [11] investigated the effects of process parameters – such as the water-to-slag ratio, number of wash cycles, stirring time, and quicklime dosage – on the ammonia-nitrogen removal rate in EMR. Thermal ammonia removal utilizes high-temperature pyrolysis to break down the stable ammonium salt structures in EMR, allowing ammonia-nitrogen to escape in gaseous form. Chen Hufei [12] calcined EMR at 750 – 1000 °C, achieving nearly complete removal of ammonia nitrogen. HE et al. found that washing EMR after calcining it at 650 °C for 30 minutes resulted in complete removal of ammonia n itrogen.

Existing studies have largely focused on optimizing parameters for a single process, lacking comparative analyses of the two processes under the same feedstock conditions. Furthermore, no multidimensional evaluation system covering process characteristics, economic costs, and resource recovery potential has been established, making it difficult to provide sufficient support for process selection by electrolytic manganese enterprises. Based on

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this, this paper uses EMR generated by an electrolytic manganese enterprise in Yichang as feedstock to conduct comparative experiments on wet and pyrometallurgical ammonia removal under identical feedstock conditions. A comprehensive evaluation is performed by integrating process characteristics, application costs, and resource recovery potential, with the aim of providing a reference for electrolytic manganese enterprises in selecting an appropriate ammonia removal process.

2. EXPERIMENTAL SECTION

2.1. Experimental materials

The EMR used in the experiment was obtained from an electrolytic manganese plant in Yichang; it had a moisture content of 25.70 % and appeared as a grayish-black, mud-like substance. The sample was crushed, sieved through a 0.4 mm sieve, dried at 100 °C, and stored in a sealed container for later use. The alkaline additive used in the wet deammonization process was commercially available calcium carbide residue with a CaO content of 65.48 %. The chemical compositions of the experimental EMR and calcium carbide slag are shown in Table 1.

Table 1. Chemical composition of EMR and calcium carbide residue (mass fraction)

Materials	N	S	Mn	Ca	Si	Al	Mg	Fe
EMR	0.74	8.26	3.25	4.17	18.86	5.23	1.34	4.03
Carbide slag	–	–	–	46.77	1.78	0.88	0.056	0.35

Fig. 1 shows the XRD analysis results for EMR. As can be seen from Table 1 and Fig. 1, EMR primarily consists of phases such as quartz and calcium sulfate dihydrate.

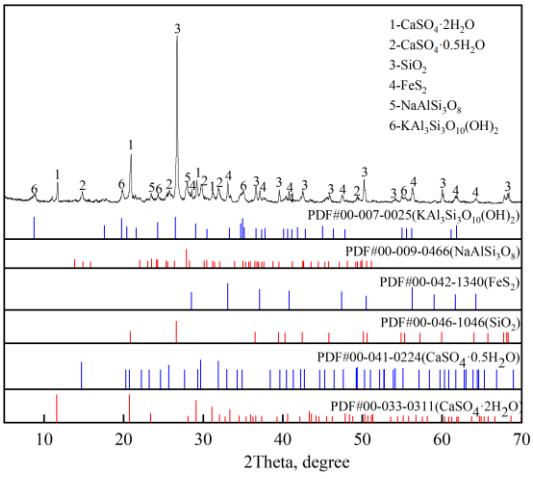

Fig. 1. XRD pattern of EMR

The diffraction peaks corresponding to each phase are relatively sharp, indicating that the mineral crystal structures are relatively intact and the degree of crystallinity is high. Although no diffraction peaks from nitrogen-containing substances were detected in the XRD pattern (possibly due to low content, poor crystallinity, or being encapsulated by other phases), the chemical analysis in Table 1 confirms that nitrogen-containing substances do indeed exist in EMR, typically in the form of $(NH_4)_2SO_4$.

Fig. 2 shows an SEM image of EMR. It reveals that the surface of EMR particles is relatively rough, the overall structure is loose, and the flake-like grains are stacked in a disordered manner, which facilitates liquid penetration or gas diffusion.

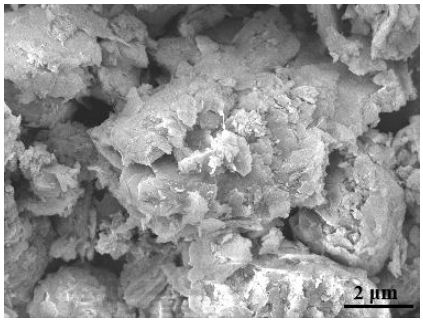

Fig. 2. SEM image of EMR

Fig. 3 shows the thermogravimetric analysis (TGA) results for EMR. It can be seen that as the temperature rises from 35 °C to 1000 °C, the total weight loss of EMR is approximately 26 %.

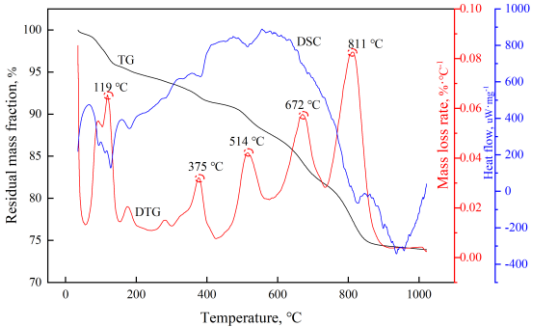

Fig. 3. TG-DTG-DSC curves of EMR

The DTG curve exhibits five distinct weight loss rate peaks; among them, the peak at 514 °C primarily corresponds to the thermal decomposition of $(NH_4)_2SO_4$.

2.2. Experimental instruments and equipment

JJ-6 High-Speed Mixer, Jintan City BoKe Testing Equipment Research Institute; SX2-16-17H High-Temperature Box-Type Resistance Furnace, Hebei Tianjian Engineering Instrument Co., Ltd.

2.3. Experimental methods

2.3.1. Wet ammonia removal experiment

An orthogonal experimental design was employed to investigate the effects of calcium carbide slag addition (mass ratio, as below), stirring time, stirring speed, and liquid-to-solid ratio (the ratio of the mass of water to the mass of solid material) on the ammonia removal rate. The experimental conditions are shown in Table 2. The specific procedure involved adding a predetermined proportion of calcium carbide slag to the EMR, adding purified water according to the liquid-to-solid ratio, stirring at the set speed for the corresponding duration, and then performing vacuum filtration. The filter cake was dried, weighed, and its nitrogen content determined, while the filtrate was measured by volume and its ammonia-nitrogen

concentration tested. The ammonia removal rate was calculated using Eq. 1.

$$R=(1-\frac{\beta\times m_1}{\alpha\times m_0})\times 100\%, \quad (1)$$

where R is the ammonia removal rate, %; β is the N content of the filter cake, %; m_1 is the mass of the filter cake, g; α is the N content of the EMR, %; m_0 is the mass of the EMR, g.

2.3.2. Pyrometallurgical ammonia removal experiments

A single-factor experimental design was employed to investigate the effects of calcination temperature and time on the ammonia removal rate. EMR was placed in a high-temperature box-type resistance furnace and calcined under an air atmosphere at heating rates of 10 °C/min to 300, 400, and 500 °C, respectively. The sample was then cooled in the furnace to room temperature, removed, weighed, and the nitrogen content was determined. At 300 °C, the samples were held for 120, 150, 180, 210, and 240 min, respectively; at 400 °C, for 90, 120, 150, 180, and 210 min, respectively; and at 500 °C, for 30, 60, 90, 120, and 150 min, respectively. The ammonia removal rate is calculated using Eq. 1, where m_1 and β represent the mass and nitrogen content of the calcined sand, respectively.

3. RESULTS AND DISCUSSION

3.1. Wet ammonia removal performance

The results of the wet ammonia removal experiments are shown in Table 2. Among the nine experimental groups, the ammonia removal rates ranged from 78.43 % to 86.01 %, with the highest value exceeding the lowest by 7.58 percentage points. Analysis of the range indicates that the calcium carbide residue addition rate had the greatest impact on the ammonia removal rate, while the effects of stirring time, stirring speed, and liquid-to-solid ratio were relatively minor. As the calcium carbide residue addition rate increased from 0 % to 20 %, the ammonia removal rate first increased rapidly and then increased only slightly; there was little difference in the ammonia removal rate between 10 % and 20 % addition rates. This is because, without the addition of calcium carbide residue, only the washing action of water caused a portion of the soluble ammonia nitrogen to enter the liquid phase; after adding 10 % calcium carbide residue, the solution's pH increased to above 10.5, and the abundant OH^- reacted with NH_4^+ to form NH_3, promoting the dissolution of more ammonia-containing nitrogen substances in the liquid and their release as NH_3; When the addition rate was further increased to 20 %, the ammonia removal rate increased only slightly because a large portion of the ammonia nitrogen had already been removed. Furthermore, the ammonia nitrogen concentrations in the filtrates from all nine experimental groups exceeded the limit specified in GB 8978-1996, "Comprehensive Discharge Standard for Wastewater" (Class I standard: 15 mg/L).

Taking into account both ammonia removal efficiency and economic costs, a calcium carbide residue addition rate of 10 %, a mixing time of 30 min, a mixing speed of 300 r/min, and a liquid-to-solid ratio of 20 were determined to be optimal. Experiments conducted under these optimized conditions yielded a ammonia removal rate of 86.35 %, with the filtrate having a pH of 11.0 and an ammonia nitrogen concentration of 273 mg/L. Since the ammonia nitrogen concentration and pH of the filtrate exceed the permissible values specified in GB 8978-1996 "Comprehensive Wastewater Discharge Standards," the filtrate cannot be directly reused or discharged. Additional facilities, such as ammonia nitrogen stripping and wastewater treatment, are required, which will significantly increase both capital investment and operating costs.

3.2. Results of pyrometallurgical de-ammonization

The results of the thermal ammonia removal experiments are shown in Fig. 4. The ammonia removal rates under different calcination conditions varied significantly; the group with the highest ammonia removal rate (96.63 %) was 79.19 percentage points higher than the group with the lowest rate (17.44 %), which is far greater than the 7.58 percentage point difference observed in wet ammonia removal. This indicates that the effectiveness of thermal ammonia removal is more sensitive to experimental parameters.

Table 2. Experimental results of wet ammonia removal

No.	Carbide slag ratio/%	Stirring time, min	Stirring speed, $r\cdot min^{-1}$	Liquid-to-solid ratio	Ammonia nitrogen concentration in filtrate, $mg\cdot L^{-1}$	N Content in filter cake, %	Ammonia removal rate, %
1	0	20	200	10	515	0.19	78.43
2	0	30	300	20	291	0.18	80.05
3	0	40	400	30	200	0.18	80.30
4	10	30	400	10	384	0.11	84.91
5	10	40	200	20	269	0.11	85.21
6	10	20	300	30	190	0.11	85.43
7	20	40	300	10	235	0.10	84.93
8	20	20	400	20	275	0.10	85.34
9	20	30	200	30	188	0.10	86.01
Mean 1	79.59	83.07	83.22	82.76	–	–	–
Mean 2	85.18	83.66	83.47	83.53	–	–	–
Mean 3	85.43	83.48	83.52	83.91	–	–	–
Range	5.83	0.59	0.25	1.16	–	–	–

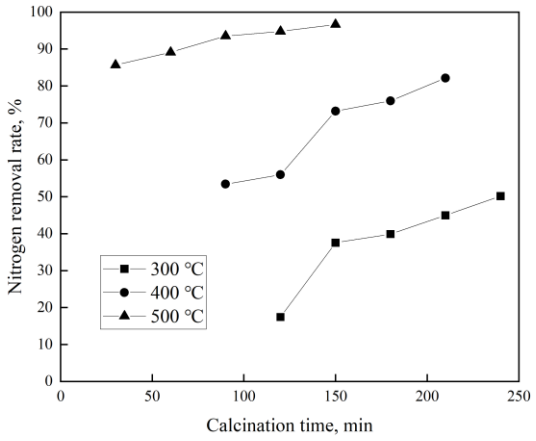

Fig. 4. Results of pyrometallurgical ammonia removal experiments

As shown in Fig. 4, both calcination time and calcination temperature have a certain influence on the ammonia removal rate, and there is an interaction between the two. On the one hand, at the same calcination temperature, the ammonia removal rate increases with longer calcination time. On the other hand, the extent to which calcination time affects the ammonia removal rate is constrained by the calcination temperature. At lower calcination temperatures (300 °C and 400 °C), extending the calcination time can significantly increase the denitration rate, whereas at higher calcination temperatures (500 °C), extending the calcination time results in a markedly smaller increase in the denitration rate. Compared to calcination time, calcination temperature has a more significant effect on the denitration rate. At a calcination temperature of 300 °C, extending the calcination time from 120 min to 240 min increased the denitration rate from 17.44 % to 50.18 %; at a calcination temperature of 400 °C, extending the calcination time from 90 min to 210 min increased the denitration rate from 53.41 % to 82.15 %; at a calcination temperature of 500 °C, even with a calcination time of only 30 min, the ammonia removal rate reached 85.69 %. The ammonia removal rate initially increased rapidly with longer calcination times, reaching 93.53 % at 90 min, and then increased slowly as the calcination time continued, reaching 96.63 % at 150 min. Based on the analysis of EMR properties in Section 1.1, it is known that the ammonia nitrogen in EMR primarily exists in the form of $(NH_4)_2SO_4$. The decomposition of $(NH_4)_2SO_4$ is a slow, endothermic process that begins around 280 °C and reaches its maximum decomposition rate at 514 °C [13]. At calcination temperatures of 300 °C and 400 °C, the kinetic rate of the $(NH_4)_2SO_4$ decomposition reaction is low; when the temperature rises to 500 °C, the thermal decomposition rate of $(NH_4)_2SO_4$ accelerates significantly; however, since some $(NH_4)_2SO_4$ is encapsulated by inert minerals such as quartz, it is difficult to remove it completely even by extending the calcination time, and thus there is an upper limit to the ammonia removal rate. Taking into account both ammonia removal efficiency and roasting energy consumption, a roasting temperature of 500 °C and a roasting time of 90 min are recommended. Under these conditions, the ammonia removal rate reaches 93.53 %, which is higher than the 86.35 % ammonia removal rate achieved by the wet ammonia removal method.

3.3. Comprehensive comparative analysis of wet and thermal ammonia removal technologies

The following comparison of wet and thermal ammonia removal is presented based on ammonia removal efficiency, process characteristics, resource recovery potential, and application costs:

The efficiency of wet ammonia removal is relatively less affected by experimental conditions, with a ammonia removal rate of 86.35 % under optimized conditions. In contrast, the effectiveness of pyrometallurgical ammonia removal is closely related to experimental conditions; in particular, the calcination temperature directly determines the upper limit of the ammonia removal rate. At 500 °C for 90 minutes, the ammonia removal rate reaches 93.53 %, and at 150 minutes, it reaches 96.63 %. The pyrometallurgical ammonia removal process has significant advantages in terms of deep ammonia removal.

The main shortcomings of the wet ammonia removal process include: 1) Ammonia removal by simple water washing is limited in effectiveness and requires the addition of alkaline reagents such as calcium carbide slag. This not only increases chemical costs and causes the filtrate pH to exceed standards but also results in low concentrations of NH_3 produced during the reaction, which are difficult to capture and can easily cause secondary pollution; 2) It generates large volumes of filtrate with high ammonia-nitrogen content, and not all of this filtrate can be returned to the production system. For filtrate that cannot be recycled, a dedicated wastewater treatment system is required, increasing both capital investment and land use; 3) The filter cake has a high moisture content, requiring an additional drying process before it can be recycled; 4) The filter cake has poor long-term stability, posing a significant risk of secondary pollution.

Thermal ammonia removal does not produce any wastewater; the NH_3 generated during calcination can be absorbed by an exhaust gas capture system and used to produce ammonia water for return to the production system. In terms of carbon emissions, the carbon emission intensity of thermal ammonia removal is generally higher than that of wet ammonia removal; however, CO_2 emissions can be reduced by using clean energy. Furthermore, calcined slag can partially replace cement, thereby reducing CO_2 emissions during cement production and offsetting the carbon footprint of thermal ammonia removal to some extent. Overall, thermal ammonia removal is more suitable than wet ammonia removal for the large-scale treatment of EMR.

The slag residue from wet ammonia removal differs little from the original slag in terms of phase structure and physicochemical properties. Its resource recovery pathways are limited, and the scale of utilization is small; further modification is required to achieve high-value utilization. In contrast, thermal ammonia removal demonstrates irreplaceable advantages in subsequent resource recovery applications. Calcination can efficiently remove ammonia nitrogen, activate the pozzolanic properties of SiO_2 and Al_2O_3 in EMR, and significantly enhance the resource recovery potential of the calcined slag. However, excessively high calcination temperatures increase energy consumption; therefore, an appropriate calcination regimen

must be determined by comprehensively considering ammonia removal efficiency, activation of pozzolanic properties, and energy consumption control.

Wet ammonia removal requires large amounts of chemicals and water, involves high investment in waste liquid treatment facilities, and has low potential for resource recovery from the post-ammonia removal residue, resulting in poor overall economic performance. Although pyrometallurgical ammonia removal entails higher equipment investment and energy consumption, it can offset part of the processing costs by recovering NH_3 to produce ammonia water for return to the electrolytic manganese production system and by using the roasted ore to manufacture high-value-added products.

In summary, pyrometallurgical ammonia removal outperforms hydrometallurgical ammonia removal in terms of ammonia removal efficiency, resource recovery, and environmental controllability, and is particularly suitable for scenarios involving large-scale processing and high resource recovery requirements; hydrometallurgical ammonia removal, on the other hand, is suitable for situations with small processing volumes and limited investment capacity.

4. CONCLUSIONS

This paper focuses on the ammonia removal performance of electrolytic manganese residue (EMR). By considering process characteristics, resource recovery potential, and application costs, it compares the wet and pyrometallurgical ammonia removal processes for EMR using the same feedstock and reaches the following conclusions:

1. Under optimized conditions – a 10 % calcium carbide slag addition, a stirring time of 30 min, a stirring speed of 300 r/min, and a liquid-to-solid ratio of 20 – the wet ammonia removal process achieved an ammonia removal rate of 86.35 %. The pyrometallurgical ammonia removal process achieved a ammonia removal rate of 93.53 % when calcined at 500 °C for 90 min.
2. Wet ammonia removal has several drawbacks, including high chemical consumption, the generation of large volumes of waste liquid containing high levels of ammonia nitrogen, and difficulties in recycling the residue after treatment. Although thermal ammonia removal has higher energy consumption, the exhaust gas it produces can be recovered and reused, and the calcined sand has significant potential for resource recovery, which can effectively reduce overall disposal costs.
3. Considering the overall ammonia removal effectiveness, resource recovery prospects, and engineering feasibility, pyrometallurgical ammonia removal is the preferred process for EMR ammonia removal treatment, particularly suited for large-scale applications with high throughput and stringent resource recovery requirements. Wet ammonia removal, however, remains applicable for small-scale operations with limited investment budgets. It is recommended that enterprises conduct a comprehensive evaluation based on their production capacity, environmental requirements, and resource recovery objectives when selecting a process.
4. The generalizability of the current findings is inherentl y constrained by the specific chemical composition an d nitrogen speciation (primarily $(NH_4)_2SO_4$) of the EMR sample sourced from Yichang. Variations in mineralogy, impurity content, or the presence of refractorynitrogen species in EMR from other regions may alter the optimal process parameters and removal efficiencies observed in this study. Therefore, future research should prioritize validation studies on EMR samples from diverse geographical sources and with varying physicochemical characteristics to verify the robustness and broader applicability of the wet and pyrometallurgical deammonification technologies evaluated herein.

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