

Article

A Complete, Sustainable Utilization Strategy: From Ferronickel Slag to High-Purity Magnesium Sulfate and Portland Cement

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Abstract

Ferronickel slag, as a major solid waste in the stainless-steel industry, poses a serious threat to the environment due to its large-scale production and low utilization rate. In this study, magnesium oxide in the ferronickel slag was leached out and converted into high-purity magnesium sulfate, while the leach residue was utilized for cement clinker production. During the complete utilization of ferronickel slag, the Mg leaching efficiency reached 90.75% and was significantly enhanced by reducing the particle size of the ferronickel slag with H₂SO₄ solution as the sole solvent. High-purity magnesium sulfate with a purity of 99.92% was prepared from the leachate through a multi-step process involving primary crystallization, purification, and secondary crystallization. The leach residue, accounting for 68.20% of the original mass, was primarily composed of 79.4 wt% SiO₂ and less than 6.1 wt% MgO and is used as a key raw material in the production of Portland cement. Sintering temperature significantly influenced the structure and properties of the resulting cement. Both the Portland clinker and cement were successfully produced at sintering temperatures of 1400 °C and 1450 °C when the leach residue was used as a primary raw material, with well-developed cementitious phases of calcium silicate and aluminate formed during calcination. The setting time, soundness, and compressive and flexural strengths of the hardened C1400 and C1450 mortars met the requirements specified in relevant standards. Through this integrated process, the overall utilization rate of the ferronickel slag reached 100%. Based on a preliminary estimate, full utilization of the annual ferronickel slag production in China could substitute at least 19.5 million tons of magnesite and 15.0 million tons of silica and reduce CO₂ emissions by 10.3 million tons.

Keywords: ferronickel slag; magnesium sulfate; leaching efficiency; purification; cement clinker



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1. Introduction

The extensive use of cement inevitably leads to significant energy consumption and environmental pollution. Continuous extraction of natural resources contributes to irreversible environmental deterioration, including biodiversity loss, global warming, climate change, vegetation degradation, ecosystem destruction, river system damage, and atmospheric dust contamination [1–5]. Consequently, the utilization of solid waste as a substitute

for natural resources has become a critical priority [6–9]. Ferronickel slag (FNS) is a solid waste residue produced from the smelting process of the alloys of nickel and iron using laterite nickel ore as the raw material [10]. According to statistical data, the production of one ton of ferronickel alloy generates approximately 14 tons of FNS, yet its comprehensive utilization rate remains as low as 12% [6]. China is a leading producer of nickel and stainless steel. Annually, more than 30 million tons of FNS is generated in the country, accounting for more than 20% of global FNS production [7]. FNS has become the fourth largest smelting residue after iron slag, steel slag, and red mud. However, the majority of this is either landfilled or stockpiled, which not only occupies extensive land areas and pollutes the ecological environment but also poses a significant threat to human health and the sustainable development of the ferronickel industry [11–13].

In order to reduce the accumulation of FNS, many methods have been used. For example, the valuable metals of Ni, Co, Cu, and Zn were recovered from FNS [14–17], and various materials were prepared, including building materials [18–20], polymers [21,22], microcrystalline glass [23], refractory materials [24], cement with metals used in production as additives [25,26], and so on. Although these current applications of FNS are technically feasible, its low activity, limited content of valuable elements, and trace harmful elements limit its efficient utilization, while relatively high production costs further constrain its development. Therefore, it is imperative to develop an environmentally friendly, cost-effective, and efficient technology to enable the large-scale utilization of FNS.

High Mg content is a characteristic of FNS, usually ranging from 15.7 to 18.7 wt% [27,28]; the Mg content of FNS in this study is about 22%. The variation primarily depends on the type of nickel laterite ore and the processing technology employed. Mg is a valuable element, and the recovery of Mg from FNS to prepare $\text{MgSO}_4 \cdot n\text{H}_2\text{O}$ (MSO) has previously been reported by our group [29], wherein MSO products meeting a 2 N (99 wt%) purity grade were successfully prepared through a leaching–crystallizing–washing process in which a mixed acid composed of 95% H_2SO_4 and 5% HNO_3 was used as the leaching agent. However, the highest reported Mg leaching efficiency reached only 83.74%, with the MgO content in the leach residue remaining as high as 9.65 wt%. Notably, the MgO content is strictly regulated in many building materials due to its potential to induce cracking caused by volume expansion [30]. Furthermore, if the leach residue occupying about 68.2% of the original FNS is not properly utilized, this would seriously impede the industrial application of this technology.

In order to solve these problems, in this study, a high-value complete utilization strategy for FNS was verified. Firstly, Mg was recovered from the FNS to prepare high-purity magnesium sulfate (HP-MSO), the market value of which is several-times [31] or even an order of magnitude higher than that of industrial-grade magnesium sulfate. HP-MSO can be used in many fields, such as in feed additives, food, medicine [32–34], and so on. Secondly, leach residue, with high silicon and low magnesium contents, can be utilized as a silicon precursor to produce Portland cement. Compared to previous studies on Mg recovery from FNS, this study achieves enhanced Mg leaching efficiency while significantly reducing the MgO content of the leach residues to a level approaching the limit value (≤ 5 wt%) specified for Portland cements [35]. The high leaching efficiency, as well as the effects of sintering temperatures on the microstructure and properties of cement clinker, and the thermodynamics, purification mechanisms of crude MSO liquor and formation mechanisms of cement phases during the sintering processes were investigated.

2. Materials and Methods

2.1. Raw Materials

The FNS was obtained from a large ferronickel plant in Beihai, China, and was generated using a typical rotary kiln-electric furnace (RKEF) and a slow cooling process. IS was sourced from an iron and steel plant via blast furnace smelting method, in Guigang, China. The main chemical compositions are presented in Table 1. The FNS is primarily composed of SiO_2 , MgO , Fe_2O_3 , and Al_2O_3 , and is characterized by high contents of Mg and Si, moderate Fe content, and low levels of Al and Ca, making it a suitable feedstock for the production of high-purity magnesium sulfate. The iron slag (IS) mainly consists of Fe_2O_3 , CaO and SiO_2 , serving as the primary resource of iron. Alumina (Al_2O_3) and lime (CaO) were used as Al and Ca resources of the clinker, while gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) was added as a setting delayer during the preparation of Portland cement. Glycerinum ($\text{C}_3\text{H}_8\text{O}_3$), absolute ethyl alcohol ($\text{C}_2\text{H}_6\text{O}_3$) and benzoic acid ($\text{C}_7\text{H}_6\text{O}_2$) were used to determine the free f-CaO content in the synthesized cement clinkers. All the chemical reagents were of analytical grade and purchased from Guangdong Guanghua Sci-Tech Co., Ltd., Guangzhou, China. Tap water and standard sand were used for the preparation of Portland cement, whereas deionized water was employed in all other experimental procedures.

Table 1. Chemical compositions of the FNS and IS used in this study.

FNS	Composition	SiO_2	MgO	Fe_2O_3	Al_2O_3	Cr_2O_3	CaO	SO_3	NiO	Σ
	Content (wt%)	52.3	36.9	4.9	3.3	0.9	0.8	0.2	0.02	99.32
IS	Composition	Fe_2O_3	CaO	SiO_2	MnO	MgO	Al_2O_3	P_2O_5	Cr_2O_3	Σ
	Content (wt%)	30.50	27.90	21.10	6.65	4.17	3.50	1.94	1.11	96.87

Figure 1 shows XRD pattern of the FNS. All the diffraction peaks can be attributed to forsterite ($\text{Mg, Fe}_2\text{SiO}_4$) phase, which corresponds to Mg_2SiO_4 , as referenced by the standard card (PDF 34-0189), with no other crystalline substance detected. Due to the similar ionic radii of Mg^{2+} (0.072 nm) and Fe^{2+} (0.078 nm) [17], partial substitution of Mg^{2+} by Fe^{2+} within Mg_2SiO_4 crystal lattice resulted in the formation of $(\text{Mg, Fe})_2\text{SiO}_4$ —a solid solution phase composed of Mg_2SiO_4 and Fe_2SiO_4 . Additionally, some Fe^{2+} may co-exist with SiO_2 in an amorphous phase. No diffraction peaks corresponding to elements such as Al, Ca, Cr and Ni were observed via the XRD measurement, consistent with their low content in the residue.

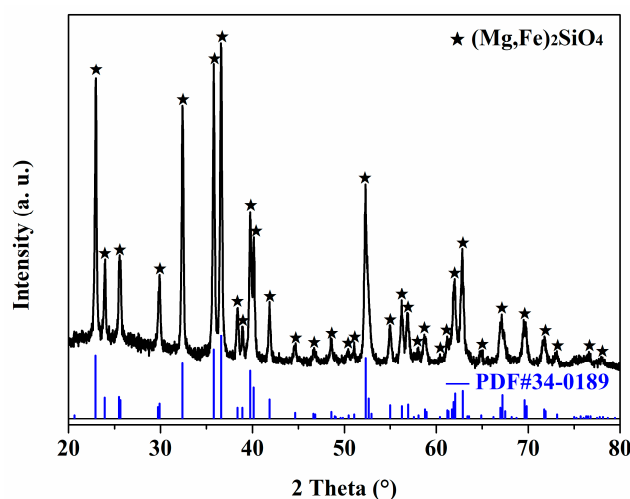


Figure 1. XRD pattern of the dried FNS powders used in this study.

2.2. Experimental Process Route

A flowchart of experimental process for the complete utilization of FNS is shown in Figure 2. The FNS, dried at 120 °C for 8 h, was subjected to ball milling in an agate jar with a ball-to-powder mass ratio of 3:1, under the following conditions: rotation speed of 600 rpm, orbital speed of 300 revolutions per minute, and milling durations of 0.25 h, 1 h, 2 h, 3 h and 6 h, respectively, followed by screening through a 200-mesh grid to obtain particles with particle sizes less than 74 µm. In the subsequent leaching experiments, 50 g of the FNS powders was loaded into a three-neck flask fitted with a reflux condenser and a thermometer. Subsequently, H₂SO₄ solution was added into the flask. The flask was placed in a mantle to facilitate the leaching of Mg from the FNS under the following reported proper conditions: liquid-to-solid ratio (L/S) 20:1, H₂SO₄ concentration (C_S) of 15 mol/L, agitation rate (r_L) of 350 rpm, leaching temperature (T_L) of 180 °C, and leaching duration (t_L) of 2 h [29]. Following the leaching process, solid–liquid separation was performed. The resulting leachate was utilized for the preparation of HP-MSO, while the leach residue served as the primary silicon source for the synthesis of Portland cement clinker. The leachate containing MSO solution was further processed to prepare HP-MSO powders by primary crystallization, purification, and secondary crystallization, while the leach residue was combined with selected mineralizers. The resulting mixture was ground at 300 rpm for 2 h and subsequently sintered at 1300 °C, 1350 °C, 1400 °C and 1450 °C for 1 h, respectively, into cement clinker. Following that, a certain proportion of lime was mixed with the clinker, and after grinding at 300 rpm for 2 h, a Portland cement was produced. Finally, the prepared Portland cement, standard sand and water were mixed, briquetted, and formed concrete after curing to test the properties of the cement.

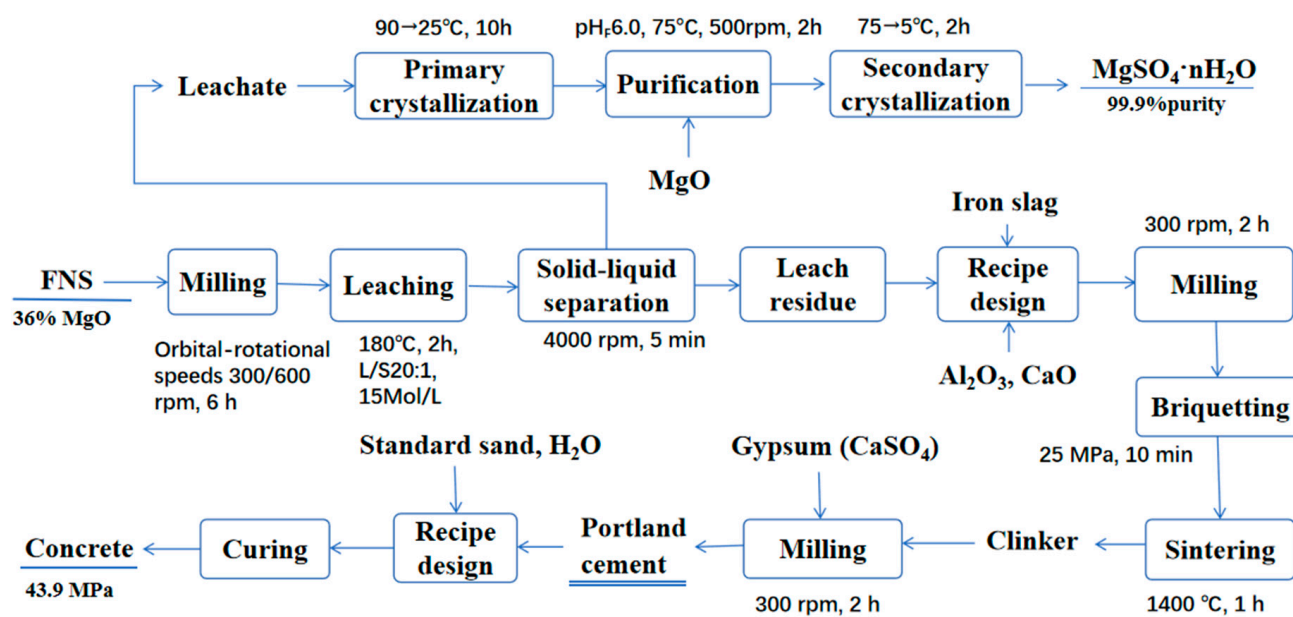


Figure 2. Experimental process flowchart for the complete utilization of FNS (the process conditions given in the figure are only the optimal ones).

2.3. Preparation Method of High-Purity Magnesium Sulfate

The leachate, obtained under the optimal leaching efficiency of Mg, was transferred to a beaker and placed in a constant-temperature low-temperature device and kept for a period of time to make magnesium sulfate crystallize slowly from the solution. After crystallization, the solid–liquid separation was carried out using vacuum filtration equipment, yielding crude magnesium sulfate heptahydrate (MgSO₄·7H₂O). The components of both

the solid and liquid phases were analyzed to determine the crystallization ratio during the primary crystallization.

To improve the product purity, the crude magnesium sulfate crystals obtained from the primary crystallization were dissolved in deionized water at a liquid–solid ratio of 3:1 mL/g to produce a crude magnesium sulfate liquor. The solution was placed on a heating platform and subjected to isothermal heating at 75 °C with an initial solution pH value (pH_I) of about 0.45, and then the neutralizing agent of magnesia (MgO) was continuously added into the liquor to adjust the pH value. During the addition, the solution was stirred at a constant speed (r_H) of 500 rpm. As the pH value increased, the liquor became cloudy and gradually formed precipitates. When the final pH value (pH_F) reached the preset level, the solution was left quietly for 4 h and then subjected to solid–liquid separation via vacuum filtration. The purified solution of magnesium sulfate and the solid residue were analyzed for their chemical components. The purified solution obtained with optimal purity was concentrated to 45% (according to the mass percent of $MgSO_4$) through heating and evaporation, cooled to 75 °C, and then transferred to the low-temperature device again at about 5 °C and kept for 2 h to finish secondary crystallization of MSO. After that, the solid–liquid separation was carried out using vacuum filtration equipment to obtain the final MSO crystal powder product. The components of the solid and the liquid phase were analyzed to determine the secondary crystallization ratio and the purity of solid products. Since magnesium sulfate heptahydrate is susceptible to decomposition or dehydration under vacuum conditions and electron beam irradiation during SEM observation, the samples were first dried at 80 °C under vacuum for 6 h and then subjected to gold coating on their surfaces prior to analysis.

2.4. Preparation Method of Portland Cement

In the experimental study on the preparation process of Portland cement clinker using the leach residues, the ratio factors of KH , SM , and IM were set as 0.82, 2.04, and 0.95, respectively, based on the values reported in the literature [36]. The relatively lower KH and IM values were selected to increase the incorporation proportion of the leach residue while reducing the required amount of lime. The recipe design for the clinker preparation is shown in Table 2, with raw materials including the FNS leach residue, blast furnace iron slag, and the chemical reagents of Al_2O_3 and CaO . These materials were mixed and dry-milled for 1 h, followed by the addition of 6 wt% water to the ground powders, and continued milling for 1 h. The ground powders were pressed into cylindrical pellets ($\Phi 50 \times 20$ mm) under a pressure of 25 MPa for 10 min. The green bodies were then dried at 100 °C for 1 h in a vacuum oven. After sintering, at temperatures of 1350 °C, 1400 °C and 1450 °C for 1 h in a muffle furnace, respectively, the sintered blocks were removed from the furnace immediately and cooled down to room temperature rapidly with an electrical fan, and finally, the blocks were ground into fine powders of Portland cement clinkers. The cement clinkers produced at 1350 °C, 1400 °C and 1450 °C were designated as C1350, C1400 and C1450 clinker, respectively.

Table 2. Recipe design of the raw materials for the preparation of Portland cement clinker.

Ratio Factors	Recipe of Raw Materials	Weight Percent (wt%)
	Leaching Residue	23.95
$KH = 0.82$	Iron slag	20.72
$SM = 2.04$	Lime (CaO)	35.08
$IM = 0.95$	Alumina (Al_2O_3)	2.26

2.5. Analysis and Calculation Method

The FNS, leach residue, HP-MSO and cement clinker were characterized using an X-ray diffractometer (XRD, Rigaku D/MAX 2500 V, Japanese Science Company, Tokyo, Japan) with Cu K α radiation at a scanning speed of 5°/min and a step width of 0.01°. Their microscopic structure was characterized with scanning electron microscopy (SEM, 800-07334, Thermo Fisher Scientific, Feiner, The Netherlands) equipped with an X-ray energy-dispersive spectrometer (EDS). The particle size distribution of the FNS powders was analyzed using a multi-angle particle size and highly sensitive Zeta potential analyzer (Nano Brook Omni, Brookhaven, GA, America). The elemental contents of the solution and crystal products were determined using inductive plasma mass spectrometry (ICP-MS, ICPS-7510, Shimadzu Company, Shimada, Japan), and the element contents of the FNS, leach residue and cement clinker were determined via X-ray fluorescence (XRF, XRF-1800, Shimadzu Company, Shimada, Japan). The *pH* value of solutions was measured using a *pH* meter (pHSJ-4F, Shanghai Instrument & Electrical Scientific Instrument Co., Ltd., Shanghai, China). The index of f-CaO was measured using a glycerol–ethylene glycol method based on international standards [37] for chemical analysis of cement. The water demands of normal consistency, setting time, and stability of the clinker were measured based on an international standard [38] using a cement paste mixer (NJ-160, Qingdao DEX Machinery Co., Ltd, Qingdao, China), a Lees clamp tester (LD-50, Qingdao DEX Machinery Co., Ltd, Qingdao, China) and a boiling box (FZ-31A, Qingdao DEX Machinery Co., Ltd, Qingdao, China).

The mixture composed of 95% clinker and 5% calcium sulphate was loaded into a planetary ball mill, thoroughly ground, and sieved through a 74 μ m mesh to produce Portland cement made of leached residue of FNS (called LR-FNS cement in this study). All composition percentages including element content and product purity in this study are reported as weight percentage (wt%) unless otherwise specified. Based on the clinkers used, the resulting cements were designated as C1350, C1400 and C1450 cement, respectively. Cement mortar was prepared by mixing the cement, standard sand, and water in a mortar mixer (NJ-160, Qingdao, China) at a mass ratio of 1:3:0.5, and after homogenization, the mixture was cast into a cuboid plastic mold with dimensions of 40 $\times$ 40 $\times$ 160 mm. After smoothing the top surface, these cuboids were put in a cement standard curing box (SHBY-40B, Qingdao DEX Machinery Co., Ltd, Qingdao, China) to cure for 3 days, 7 days and 28 days, respectively, under controlled conditions of 20 $\pm$ 1 °C temperature and 90% relative humidity. Finally, these hardened cuboids were taken out from the box and removed from the molds to test their mechanical properties in accordance with the international standard [39].

The leaching efficiency (η_L) of the elements (Mg, Fe, Al, Ca, Cr, Ni) from FNS after leaching was calculated according to Equation (1), while the residue ratio (γ) during the leaching step was calculated according to Equation (2).

$$\mu_L = \frac{cV}{w_0m_0} \times 100\% \quad (1)$$

$$Y = \frac{m'_0}{m_0} \times 100\% \quad (2)$$

where *c* is the content (g/L) of elements (Mg, Fe, Al, Ca, Cr and Ni) in the leachate, *V* is the volume (L) of the leachate, *m*₀ and *w*₀ are, respectively, the mass of FNS (g) and the mass percent of element in the FNS (mas%), and *m*'₀ is the mass of the leach residue (g).

Crystallization ratio (ψ_c) of the magnesium sulfate during primary and secondary crystallization was calculated using Equation (3).

$$\psi_c = \frac{c_0 V_0 - c'_0 V'_0}{c_0 V_0} \times 100\% \quad (3)$$

where c_0 and c'_0 are the content (g/L) of elements in the liquor before and after crystallization, respectively, and V_0 and V'_0 are the volume (L) of the liquor before and after crystallization, respectively.

The removal ratio (Φ_R) of the main impurity elements and the Mg loss ratio (Φ_{Mg}) during the purification process were calculated according to Equations (4) and (5), respectively.

$$\Phi_R = \frac{c_1 V_1 - c'_1 V'_1}{c_1 V_1} \quad (4)$$

$$\Phi_{Mg} = \frac{c_1 V_1 + qw\beta - c'_1 V'_1}{c_1 V_1 + qw\beta} \quad (5)$$

where c_1 and c'_1 denote the content (g/L) of elements, V_1 and V'_1 denote the volume (L) of the liquors before and after purification, respectively, q denotes the mass (g) of the neutralizer MgO, w and β denote the mass percentage (%) of Mg in one MgO molecule and the purity (%) of the neutralizer MgO, respectively.

To ensure reliability, all experiments for key metrics, such as elemental leaching efficiency, sample purity, impurity removal rate, loss rate, crystallization efficiency, and cement and clinker performance, were performed at least in triplicate. The average of these replicates was used as the final measured result. Error bars in both dot-line and bar graphs represent the range of experimental variation.

3. Results and Discussion

3.1. Performance Analysis of Pretreated FNS

The changes in the microscopic morphology of the FNS ground for various milling durations t_m were observed via SEM. Figure 3a–c show that the ground FNS particles displayed irregular shapes with sharp edges and corners, and the mean size gradually decreased with increasing t_m . After milling for 6 h, particles larger than 5 μm became rare. The particle size distribution results for the ground FNS powders are shown in Figure 3d. The mean particle size decreased from 1.83 μm to 0.78 μm as t_m increased from 0.25 h to 6 h, and the particle size trend is consistent with SEM observation. The reduced particle size of FNS powders enhanced the leaching process and improved Mg leaching efficiency.

3.2. Results and Analysis of Leaching Process

It should be emphasized that the leaching experiments in this study focused solely on the influence of the milling durations on the leaching efficiency of relevant elements in FNS and did not evaluate the effects of leaching conditions such as sulfuric acid concentration, leaching temperature, and leaching duration, as these factors have been thoroughly investigated in previous studies [29]. The leaching conditions employed in this study were selected based on previously established optimal values. The chemical components of the leachates, obtained after liquid–solid separation following the leaching of FNS ground for various durations, are shown in Table 3. The content of Mg and Fe was much higher than that of Cr, Al, Ca and Ni in the leachates for all the leaching experiments. Compared with the leachate from the FNS ground for 0.25 h, the Mg content in the leachate obtained from the FNS ground for 6 h increased from 7.2968 g/L to 9.1332 g/L. However, the content of primary impurity, Fe, also increased correspondingly from 0.8814 g/L to 1.0050 g/L.

The levels of the other impurities were much lower than those of Fe, confirming Fe is the predominant impurity in the MgSO_4 leachates under all experimental conditions.

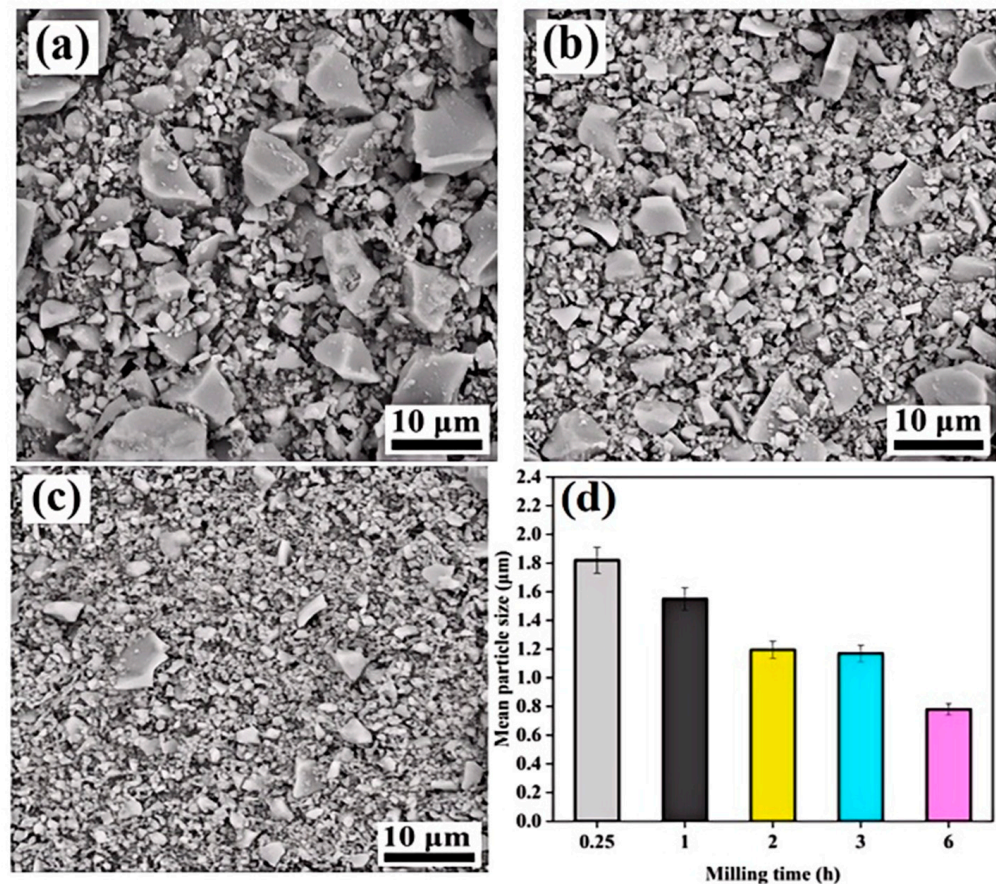


Figure 3. SEM micrographs of the FNS experienced various milling durations (t_m) of (a) 0.25 h, (b) 3 h, (c) 6 h, respectively, and (d) the effect of t_m on the mean particle size of the ground FNS.

Table 3. The content of Mg and relevant metal elements in the leachates obtained through a liquid–solid separation after leaching, in which the FNS ground at various t_m was used as solute under the following leaching conditions: $T_L = 180\text{ }^\circ\text{C}$, $t_L = 2\text{ h}$, $L/S = 20:1$, $C_S = 15\text{ mol/L}$, $r_L = 350\text{ rpm}$ (g/L).

Elements	Mg	Fe	Cr	Al	Ca	Ni
$t_m = 0.25\text{ h}$	7.2968	0.8814	0.1030	61.8541 *	13.0459 *	3.4164 *
$t_m = 1\text{ h}$	7.9505	0.9618	0.1123	64.7895 *	13.2923 *	4.3073 *
$t_m = 2\text{ h}$	8.2300	0.9732	0.1129	68.2159 *	13.4375 *	4.1709 *
$t_m = 3\text{ h}$	8.3782	0.9759	0.1144	68.2895 *	13.9982 *	4.4123 *
$t_m = 4\text{ h}$	8.5750	0.9855	0.1176	71.2514 *	14.5427 *	4.2245 *
$t_m = 6\text{ h}$	9.1332	1.0050	0.1205	78.7073 *	14.8445 *	5.8800 *

Note: * represents the unit of mg/L.

The effects of t_m on the leaching efficiency of elements are shown in Figure 4a. The overall trend indicated a gradual increase in the elemental concentrations in the leachates with the prolongation of t_m , because the total contact area between the solute and solvent increased owing to the decrease in particle size for the FNS during the leaching step, resulting in more complete leaching. The leaching efficiency of Mg increased significantly from 72.51% to 90.75%, whereas that of Fe, Al, Ca, Cr and Ni exhibited relatively minor

changes as the t_m increased from 0.5 h to 6 h. The low Ca content in the leachates is probably attributed to the low solubility of CaSO_4 in the high-acid and high-temperature solution system and the common-ion effect resulting from high Mg^{2+} content. It has been reported that the solubility of CaSO_4 is only 7.79 g per 100 g water in 95.9% H_2SO_4 solution at a leaching temperature of 180 °C [40]. The high content of Mg^{2+} further suppressed the solubility of CaSO_4 in the leachates due to the common-ion effect. As a result, the solubility of CaSO_4 decreased with the increase in the MgSO_4 content, and a similar phenomenon was observed in a solution system of 0.3 M H_2SO_4 -0.06 M NiSO_4 -0.005 M $\text{Al}_2(\text{SO}_4)_3$ -0.1 M MgSO_4 [41]. The results of leaching experiments show that it is appropriate to choose a 6 h milling duration for FNS pretreatment. Figure 4b illustrates the relationship between t_m and the residue ratio (γ). γ decreased from 72.60% to 68.20% with the prolongation of t_m from 0.25 h to 6 h, which is consistent with the trend of the Mg leaching efficiency, which serves as an important indicator of the extent of solid waste reduction.

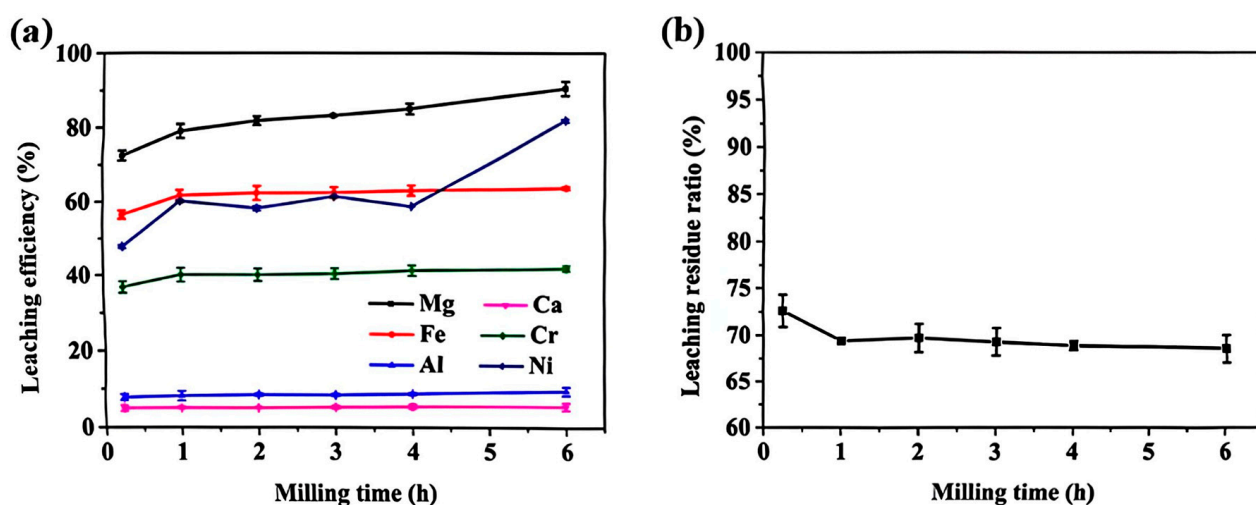


Figure 4. (a) The effect of t_m on the leaching efficiency of Mg and relevant metal elements; (b) the relationship between residue ratio (γ) and t_m under the same leaching conditions.

Table 4 shows the chemical components of the leach residue. Based on the calculation of element mass balance, the majority of Mg entered into the leachate, whereas most of Al and Ca remained in the leach residue. Compared to the chemical compositions of the FNS (Table 1), this leach residue, characterized by high SiO_2 and low MgO contents, is more suitable as a raw material for building products.

Table 4. Chemical components of the leaching residue obtained from the FNS milled at 6 h.

Components	SiO_2	MgO	Al_2O_3	Fe_2O_3	SO_3	CaO	Cr_2O_3	Σ
Content (wt%)	79.4	6.1	4.4	4.1	3.9	1.3	0.7	99.9

Figure 5a–c show the SEM-EDS results of the leaching residue obtained from the FNS milled for various t_m . The leach residue consists of many irregularly shaped particles with a broad size distribution, and these particles appear dense and lack pores, even on the coarse particles. The EDS results indicate that, with the prolongation of t_m , the content of Mg in the leach residue gradually decreased, while the content of Si increased, and the trend is consistent with the Mg leaching efficiency results.

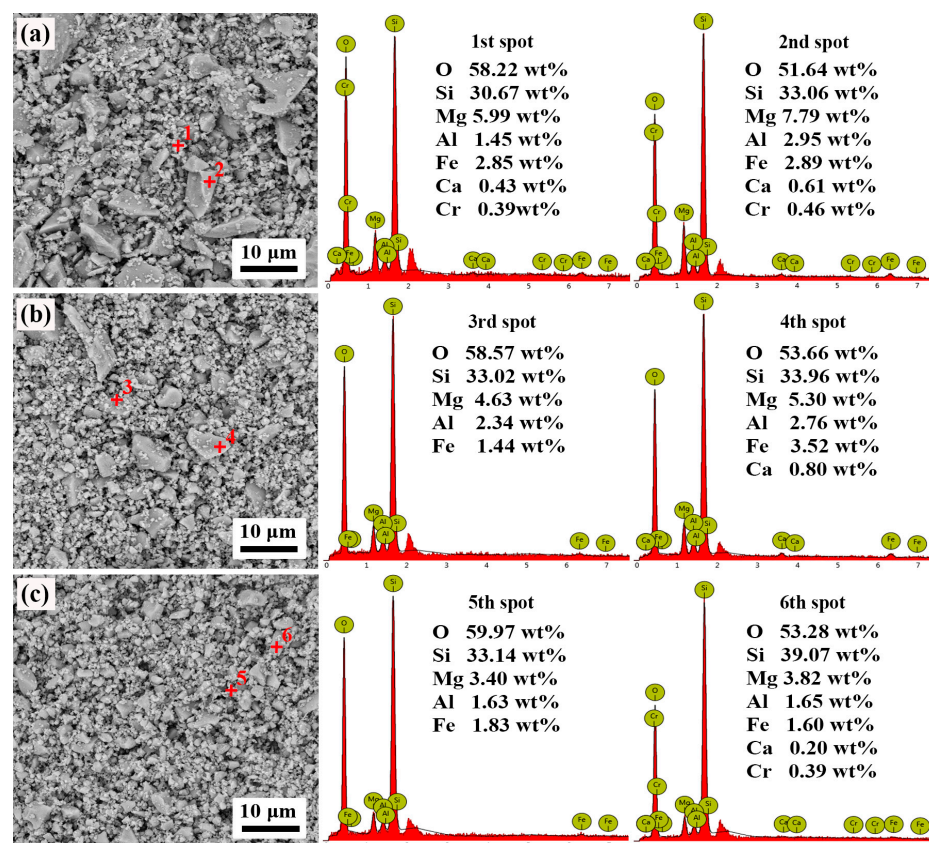


Figure 5. SEM-EDS results of the leach residue obtained from the FNS milled at various t_m of (a) 0.25 h, (b) 3 h, (c) 6 h, under the same leaching conditions.

Figure 6 shows XRD results for the leach residue obtained from the FNS with various t_m . The dominant crystalline phase in these samples remained as an olivine ($\text{Mg, Fe})_2\text{SiO}_4$ phase, and no other crystalline phases were observed as the sulfates of Mg, Fe and Al produced from the reactions of FNS and H_2SO_4 are soluble substances and entered into the leachate in the state of ions during the leaching process, and the other compounds were not detected due to their low contents. A broad diffraction bulge between 20° and 30° corresponds to amorphous silica [42,43]. The bulges became more apparent with increasing t_m because much more amorphous silica was produced and precipitated into the leach residue for the FNS with smaller particles. Thermodynamically, Mg_2SiO_4 reacted with H_2SO_4 to produce H_2SiO_3 , which was readily decomposed into amorphous silica under a leaching temperature of 180°C , due to its instability [29]. As the prolongation of t_m increased from 0.25 h to 6 h, the intensity of the diffraction peak in the XRD pattern decreased gradually, resulting from the progressive disruption of the $(\text{Mg, Fe})_2\text{SiO}_4$ lattice by Mg^{2+} release during the leaching process.

3.3. Results and Analysis of the Primary Crystallization

To investigate the effects of process conditions on the impurity removal ratio and purity of the products, the leachate used for preparing the HP-MSO product was exclusively derived from the leaching processes of FNS ground for 6 h. The primary crystallization conditions were based on the previously reported optimal values [29], including an initial crystallized temperature (T_{PI}) of 90°C , a final crystallized temperature (T_{PF}) of 25°C , a crystallized time (t_P) of 10 h, and an agitation rate (r_P) of 50 rpm. Due to variations in the composition of the leaching solution, even under the same crystallization conditions, the crystallization efficiency and MSO purity differ from the previous results. The results of primary crystallization are shown in Figure 7, where the Mg crystallization ratio reached

85.51%, indicating that most Mg was successfully crystallized from the solution and effectively separated from the elements of Al, Ca and Cr due to their low initial content and crystallization efficiencies. However, the impurities of Fe and Ni showed a relatively high crystallization ratio, suggesting their tendency to co-crystallize with Mg.

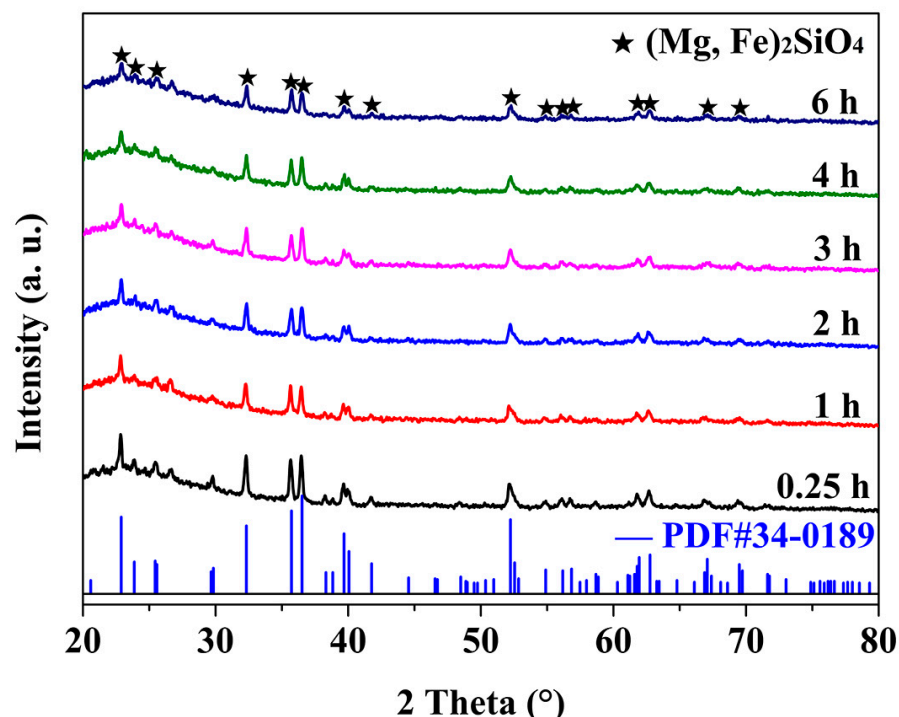


Figure 6. XRD pattern of the leach residue obtained from the FNS milled at various t_m under the same leaching conditions.

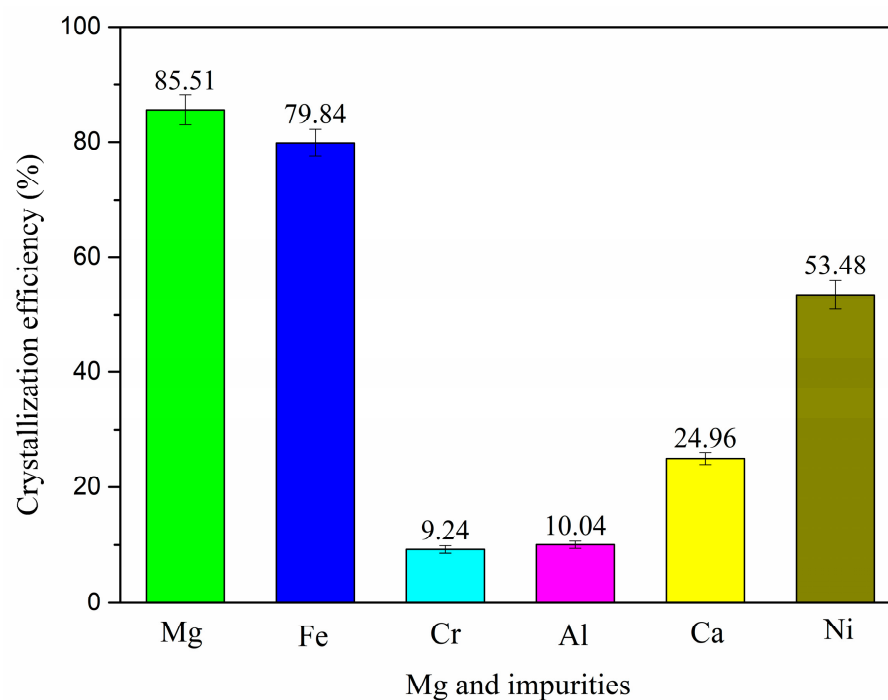


Figure 7. Crystallization ratio of Mg and relevant elements in the liquor after the primary crystallization step ($T_{PI} = 90\text{ }^{\circ}\text{C}$, $T_{PF} = 25\text{ }^{\circ}\text{C}$, $t_P = 10\text{ h}$, $r_P = 50\text{ rpm}$).

The content of Mg and related elements in the crude MSO product obtained from the primary crystallization step and in the final product of HP-MSO through secondary crystallization is shown in Table 5. The Mg content in the crude MSO product is 8.21%, with Fe, Al, Cr and Ni as the main impurities. The purity of magnesium sulfate, calculated based on the content of Mg, is only 83.30%, indicating the presence of a large amount of additional impurities in the crude MSO products. The undetected main impurities are likely residual concentrated sulfuric acid solution absorbed onto the surfaces or in the pores of the crude MSO crystal particles, and the impurities were retained mainly due to the highly acidic environment with multiple impurities during the primary crystallization process. These impurities originated from three sources. First, some impurities existed as free ions in the liquid adsorbed at grain boundaries and microvoids, and most of them can be removed by washing, except those located in closed pores. Second, impurities incorporated into the crystal lattice of the MSO accounted for a relatively small proportion and could not be removed by non-destructive washing methods. The crude MSO products cannot be directly washed with water because of the high solubility of MSO in aqueous media, and, therefore, alternative washing processes, such as ethanol or supersaturated MSO solution, were rather inconvenient [29,44]. This crude product with such low purity cannot be used directly without further treatment. Although cleaning can significantly improve the purity of the MSO product, it remains ineffective in producing high-purity products. To eliminate the need for a washing step in the preparation of HP-MSO products, an additional purification step is required to remove the impurities including concentrated sulfuric acid and other impurities in the crude MSO products.

Table 5. The content of Mg and related elements in the crude MSO product obtained through the primary crystallization and the final HP-MSO product obtained through the secondary crystallization.

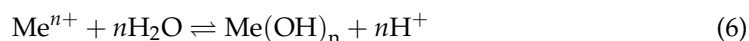
Elements	Mg	Fe	Ca	Cr	Al	Ni
Content in crude MSO product (wt%)	8.21	0.53	76.96 *	74.13 *	52.61 *	20.93 *
Content in final HP-MSO product (wt%)	9.85	35.93 *	34.52 *	3.64 *	12.10 *	4.88 *

Note: * represents the unit of mg/kg.

3.4. Results and Analysis of Purification Process

3.4.1. Thermodynamic Analysis of Purification Process

Based on the chemical component of the crude liquor, the general chemical formula of the neutralization–hydrolysis reactions during the purification process is given as Equation (6).



where Me represents metal elements of Fe, Al, Ca, Cr, Ni and Mg.

For the hydrolysis reactions of Me^{n+} , the pH value beginning to precipitate is tightly related with the solubility constant (K_{sp}) of the precipitated $\text{Me}(\text{OH})_n$, water ion constant (K_w) and the activity of Me^{n+} ($\alpha_{\text{Me}^{n+}}$), and their relationship is described by Equations (7) and (8) [36].

$$pH = \frac{1}{2.303n} [\ln K_{sp} - n \ln K_w - \ln \alpha_{\text{Me}^{n+}}] \quad (7)$$

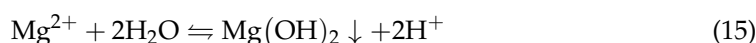
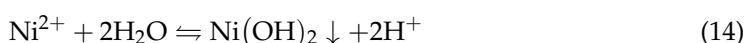
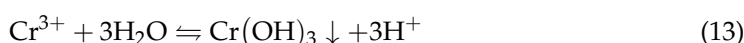
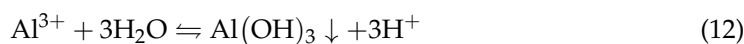
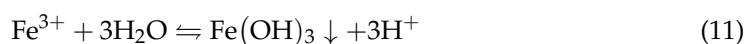
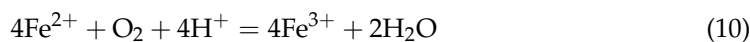
$$\ln K_{sp} = -\frac{\Delta_r G_m^\ominus}{RT} \quad (8)$$

where $\Delta_r G_m^\ominus$ is the standard Gibbs free energy change in the reaction (kJ/mol), R is the thermodynamic constant equal to 8.314 J/(mol·K), and T is the temperature (K).

In such an aqueous solution, the activity of impurity metal elements is approximately equal to their molar concentration. At 25 °C, K_w is 1.0×10^{-14} , and K_{sp} is as

follows: $[\text{Mg}(\text{OH})_2] = 6.0 \times 10^{-10}$, $[\text{Fe}(\text{OH})_2] = 8.0 \times 10^{-16}$, $[\text{Fe}(\text{OH})_3] = 3.0 \times 10^{-39}$, $[\text{Al}(\text{OH})_3] = 1.3 \times 10^{-33}$, $[\text{Cr}(\text{OH})_3] = 6.3 \times 10^{-31}$, $[\text{Ni}(\text{OH})_2] = 2.0 \times 10^{-15}$, and $[\text{Ca}(\text{OH})_2] = 1.96 \times 10^{-13}$ [45]. Based on these data and Equation (7), the theoretical hydrolysis pH values of Me^{n+} at various concentrations were calculated. Figure 8a shows the relationship between the equilibrium content of Me^{n+} and theoretical hydrolysis pH . The Me^{n+} content in this system decreases linearly with increasing solution pH , and under identical pH conditions, the precipitation of hydroxide precipitation follows the order: $\text{Fe}(\text{OH})_3 > \text{Al}(\text{OH})_3 > \text{Cr}(\text{OH})_3 > \text{Fe}(\text{OH})_2 > \text{Ni}(\text{OH})_2 > \text{Ca}(\text{OH})_2 > \text{Mg}(\text{OH})_2$. Moreover, the pH required for the complete removal of Fe^{2+} and Ni^{2+} approaches the onset precipitation pH of Mg^{2+} . Hence, to suppress the co-precipitation of Mg , iron should be maintained in the trivalent state (Fe^{3+}). In practice, Fe predominantly existed as Fe^{3+} rather than Fe^{2+} in the leachate due to the harsh leaching conditions (high temperature, high acid and intense agitation), which strongly favor Fe^{2+} oxidation. For Ca^{2+} and Ni^{2+} , their theoretical hydrolysis pH is close to that of Mg^{2+} when present at comparable concentrations, but under optimized hydrolysis conditions, Ni^{2+} could form a precipitate almost completely, causing an increase in Mg loss. Ca is the most difficult to separate completely from Mg^{2+} through a neutralization–hydrolysis method, and fortunately, its content in the MSO liquor was inherently low, rendering it less problematic. Furthermore, the onset pH for Mg^{2+} precipitation shifted to lower pH values as Mg^{2+} content in the solution increased during the purification process. To decrease Mg loss and consumption, the final pH value of the solution must be tightly controlled near 7.0.

According to the chemical components of the crude MSO liquor, the following chemical reaction equations were proposed for the purification process, in which MgO served as the neutralizer.



Equations (9)–(15) represent the main chemical reactions occurring during the purification process of the crude MSO solution. The change in Gibbs free energy (ΔG) was examined to evaluate the feasibility and direction of these reactions, with the calculated results shown in Figure 8b, obtained using HSC Chemistry 6.0 software. The ΔG values for Equations (9) and (10) are negative across the temperature range of 25–105 °C, confirming that both reactions are thermodynamically possible within the experimental purification temperature window (45–95 °C). This further demonstrates that Fe^{2+} in the crude MSO leachate is unstable and can be oxidized into Fe^{3+} under atmospheric conditions, particularly under the leaching conditions involving high temperature and intense agitation. In contrast, the ΔG values of Equations (11)–(15) are positive between 25 and 105 °C, indicating that these reactions are impossible to occur spontaneously under the applied purification conditions. Crucially, during purification, the gradual addition of MgO continuously consumes H^+ , thereby decreasing solution acidity and shifting the reaction equilibrium of Equations (11)–(15) toward the right, i.e., favoring hydroxide precipitation.

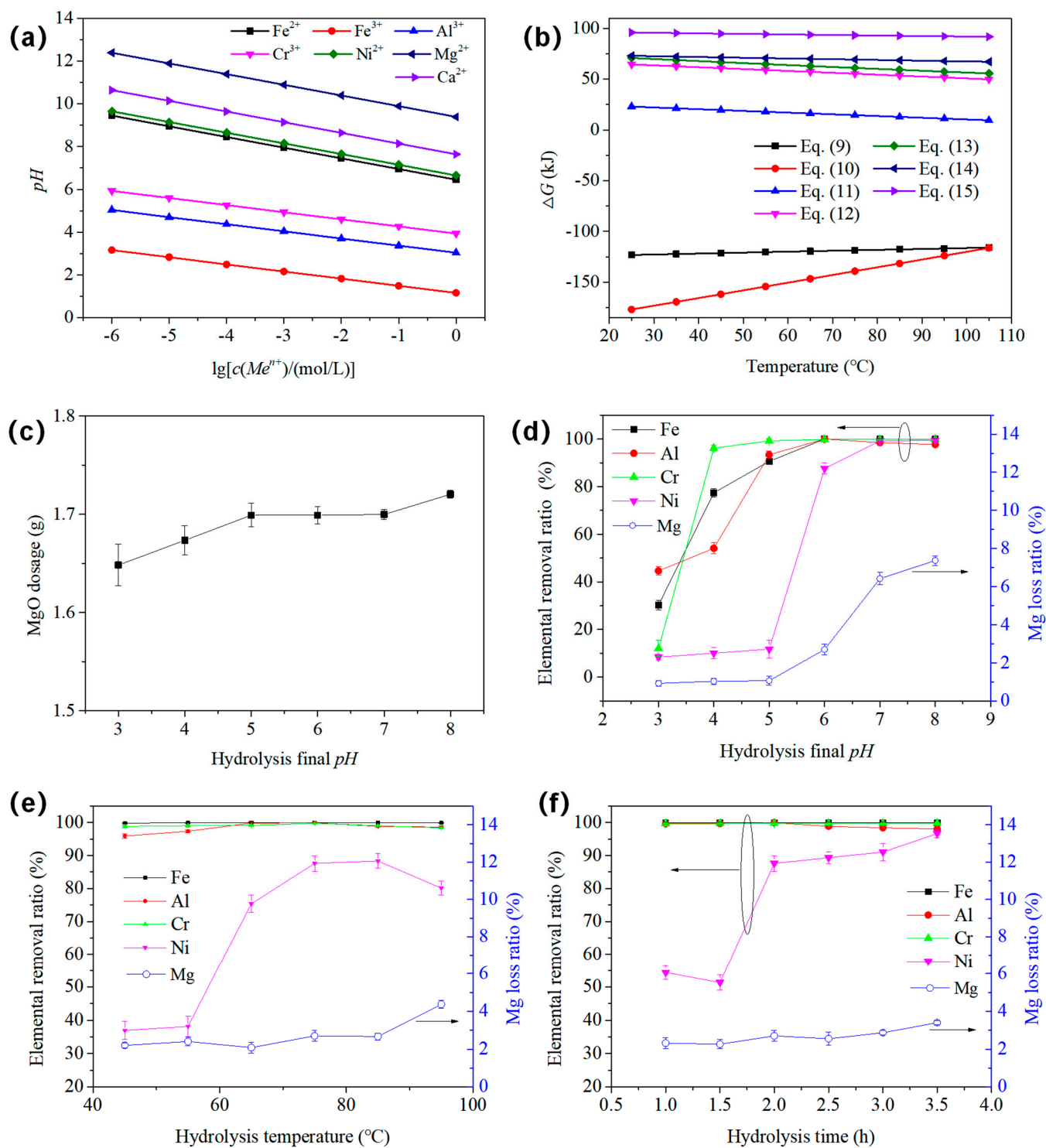


Figure 8. Relationships (a) between the content of related Me^{n+} in the liquor and the theoretical hydrolysis pH at 25 °C, (b) between ΔG of the main chemical reactions and temperature for Equations (9)–(15), and (c) between MgO dosage and pH_F . Effects of (d) pH_F ($pH_I = 0.45$, $T_H = 75$ °C, $t_H = 2$ h, $r_H = 500$ rpm), (e) T_H ($pH_I = 0.45$, $pH_F = 6.0$, $t_H = 2$ h, $r_H = 500$ rpm), and (f) t_H ($pH_I = 0.45$, $pH_F = 6.0$, $T_H = 75$ °C, $r_H = 500$ rpm) on removal ratio of impurities and Mg loss ratio during the purification step.

3.4.2. Effect of pH of Solution on Precipitation of Impurities

Figure 8c illustrates the relationship between the final pH (pH_F) of the solution and the dosage of MgO neutralizer. In the initial stage of pH 3–5, the pH of the solution increased gradually with MgO addition, as a large excess of H^+ necessitates substantial neutralizer input for effective acid consumption. As the neutralization proceeds, the content of H^+ in the solution declined rapidly, and consequently, a lower dosage of MgO will lead to significant changes in the pH of the solution. Figure 8d shows the effect of pH_F on the removal ratio of impurity elements from the crude MSO solution. When the pH_F increased from 3 to 6, the removal ratios of Fe, Al and Cr rose sharply from 30.24%, 44.63%, and 12.01% to 99.99%, 99.96% and 99.82%, respectively, whereas the loss ratio of Mg increased less clearly. With a further increase in pH_F (>6), the removal ratios of Fe and Cr gradually tended to a stable level, but the loss ratio of Mg became obvious, and the removal ratio of Al exhibited a slight decrease, probably caused by the partial re-dissolution of the $Al(OH)_3$ precipitates under strongly alkaline conditions [46]. The presence of Al in the Ni-bearing solution had a significant effect on the precipitation of Ni [47]. The Ni removal ratio was very low when $pH_F \leq 5$ but increased sharply from 11.74% to 99.19% with the increase in pH_F from 5 to 7 and then tended to be stable. The actual pH of Ni precipitation was relatively lower than the theoretical value, probably caused by the co-precipitation of Ni with trivalent metal cations of Fe^{3+} , Al^{3+} and Cr^{3+} [48]. Relevant studies revealed that the adsorption or co-precipitation of divalent transition metal elements on Fe, Al and Cr hydrated oxide precipitates is a potential way to remove heavy metals from aqueous solutions at a lower pH than those required for their direct hydroxide precipitation [47–50]. Based on holistic optimization balancing the MgO consumption, purification effect and heavy metal content, suitable purification conditions were established as follows: $pH_I = 0.45$, $pH_F = 7.0$, $T_H = 75\text{ }^\circ\text{C}$, $t_H = 2\text{ h}$, $r_H = 500\text{ rpm}$. Under these conditions, the overall removal ratio of impurities reached about 99.85%, while the Mg loss ratio was controlled below 7.00%.

3.4.3. Effect of Hydrolysis Temperature on Precipitation of Elements

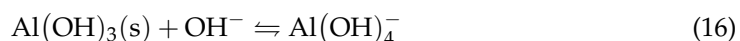
Figure 8e shows the effect of hydrolysis temperature (T_H) on the removal ratio of impurity elements and the loss ratio of Mg. Compared to those in the crude solution, the contents of Fe, Al, Cr and Ni in the purified solution exhibited a significant change with the increase in T_H , whereas the content of Mg in the solution remained relatively stable. The removal ratios of the Fe, Al, Cr and Ni first increased and then decreased. In the checked temperature range, the removal ratio of Fe, Al and Cr reached 99.90%, 95.94% and 98.44%, respectively, while the Mg loss ratio remained below 5%. In comparison, Ni precipitation is more sensitive to T_H : the removal ratio increased from 36.95% at $45\text{ }^\circ\text{C}$ to 88.35% at $85\text{ }^\circ\text{C}$, and after that, it began to decline. From a kinetic perspective, the average kinetic energy of molecules and the probability of collision and bonding increase with the increase in temperature, thereby promoting hydrolysis reactions. As indicated by the thermodynamic calculations shown in Figure 8b, the hydrolysis reactions of both Mg and impurity elements are endothermic, indicating that it is beneficial to promote the reactions and make the impurity elements hydrolyze more thoroughly through the increase in T_H . However, these hydrolysis reactions are all reversible. When the T_H becomes excessively high, the enhanced reactivity of H^+ and reduced stability of hydroxides in the purified system intensified the reverse dissolution process, resulting in a decline in the removal ratios of Ni, Al and Cr. Furthermore, elevated hydrolysis temperatures result in higher energy consumption and increasing production costs. Considering this comprehensively, a hydrolysis temperature of $75\text{ }^\circ\text{C}$ is deemed optimal, since most of Fe, Al, Cr and Ni can be precipitated from the crude MSO solution, while the loss of Mg is acceptable.

3.4.4. Effect of Hydrolysis Time on Precipitation of Elements

Figure 8f presents the effects of varying hydrolysis times (t_H) on the removal ratio of impurity elements and Mg loss ratio for the crude MSO solution. The elements of Fe, Al and Cr showed a high removal ratio close to 100% while the loss ratio of Mg was below 3.50% in the whole checked range of t_H , indicating highly effective removal of the primary impurities. As t_H increased, the content of Mg, Fe, Al and Cr elements exhibited only minor fluctuations, but Ni exhibited a distinct trend from these elements. The factor of t_H exerted a significant influence on the precipitation of Ni: its removal ratio rose sharply from 51.31% to 96.66% with prolonged t_H . Considering both the purification efficiency and residual impurity level of the solution comprehensively, an appropriate t_H range of 2.0–2.5 h is recommended for the purification process.

3.4.5. Mechanism Analysis of Purification Process

According to a systematic analysis, solution acidity is the dominant factor influencing the separation of Fe, Al, Cr and Ni from the crude MSO solution via the neutralization–hydrolysis method, and MgO addition provides an effective means to modulate this parameter. The hydrolytic precipitation behavior of these metal ions and the concurrent evolution of solution pH are thermodynamically controlled by the chemical reactions in Equations (9)–(16). Specifically, MgO reacts with H^+ in the solution according to Equation (9), thereby consuming acid and elevating the solution pH . This pH increase directly enhances the hydroxide ion activity ($[OH^-]$), which drives the hydrolysis and subsequent precipitation of impurity cations, as described by Equations (11)–(15). As MgO dosage increased, the reaction in Equation (9) proceeded towards the positive direction, leading to a rise in solution pH , which promoted the hydrolysis and precipitation of more impurities such as Fe, Cr and Ni. $Al(OH)_3$ is an amphoteric compound, and its precipitates formed via Equation (12) could redissolve via Equation (16) when the solution pH exceeded a certain threshold, entering a highly alkaline environment.



3.5. Results and Analysis of Secondary Crystallization

Solution crystallization is a phase separation process wherein solute precipitates from a homogeneous solution, which consists of two primary stages: nucleation and crystal growth. The thermodynamic driving force for both stages is solution supersaturation, which is strongly dependent on temperature-dependent solute solubility. For MSO, aqueous solubility exhibits an unusual non-monotonic trend: it increases with temperature from 0 to 75 °C, reaches a maximum near 75 °C, and then decreases from 75 °C to 100 °C [51]. Consequently, cooling crystallization was implemented over the temperature interval of 75 °C (initial) to 5 °C (final). As the solution cooled, it became supersaturated and traversed progressively higher supersaturation levels, thereby triggering spontaneous nucleation and sustained crystal growth. The primary chemical reaction governing this crystallization is shown as Equation (17).

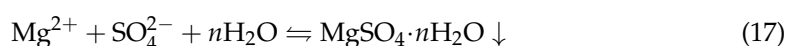


Figure 9a presents a photograph of the synthesized HP-MSO product, revealing needle-like crystallites with dendritic, snowflake-like morphologies. The microscopic structure after drying is shown in Figure 9b, revealing that the material is composed of micron-sized or sub-micron-sized crystalline grains. The XRD pattern in Figure 9c exhibited a single phase exclusively matching the monoclinic structure of $MgSO_4 \cdot 7H_2O$ (PDF#72-0696), with no additional crystal phases detected. Thermodynamic data confirmed that $MgSO_4 \cdot 7H_2O$

was more favorable for crystallization than lower hydrates ($\text{MgSO}_4 \cdot n\text{H}_2\text{O}$, $n < 7$) under a lower final crystallization temperature [29]. Figure 9d plots the crystallization ratio during the secondary crystallization, calculated according to Equation (3). The purity of the HP-MSO products was quantified via ICP-MS measurement of total Mg content, yielding a crystallization ratio of 78.01% and a chemical purity of 99.92%. A comparative analysis of the purity between HP-MSO and magnesium sulfate is provided in Supplementary Table S1, demonstrating that the product obtained in this study achieves the highest purity. In future industrialization, the post-crystallized solution will be recycled as a solvent for dissolving crude MSO, meaning that this portion of Mg retained in the post-crystallized solution constitutes recoverable inventory, not irreversible loss. Given its exceptional purity, the resulting MSO products can be classified as high-purity grade, although, currently, no international or national standards specifically define the purity for HP-MSO.

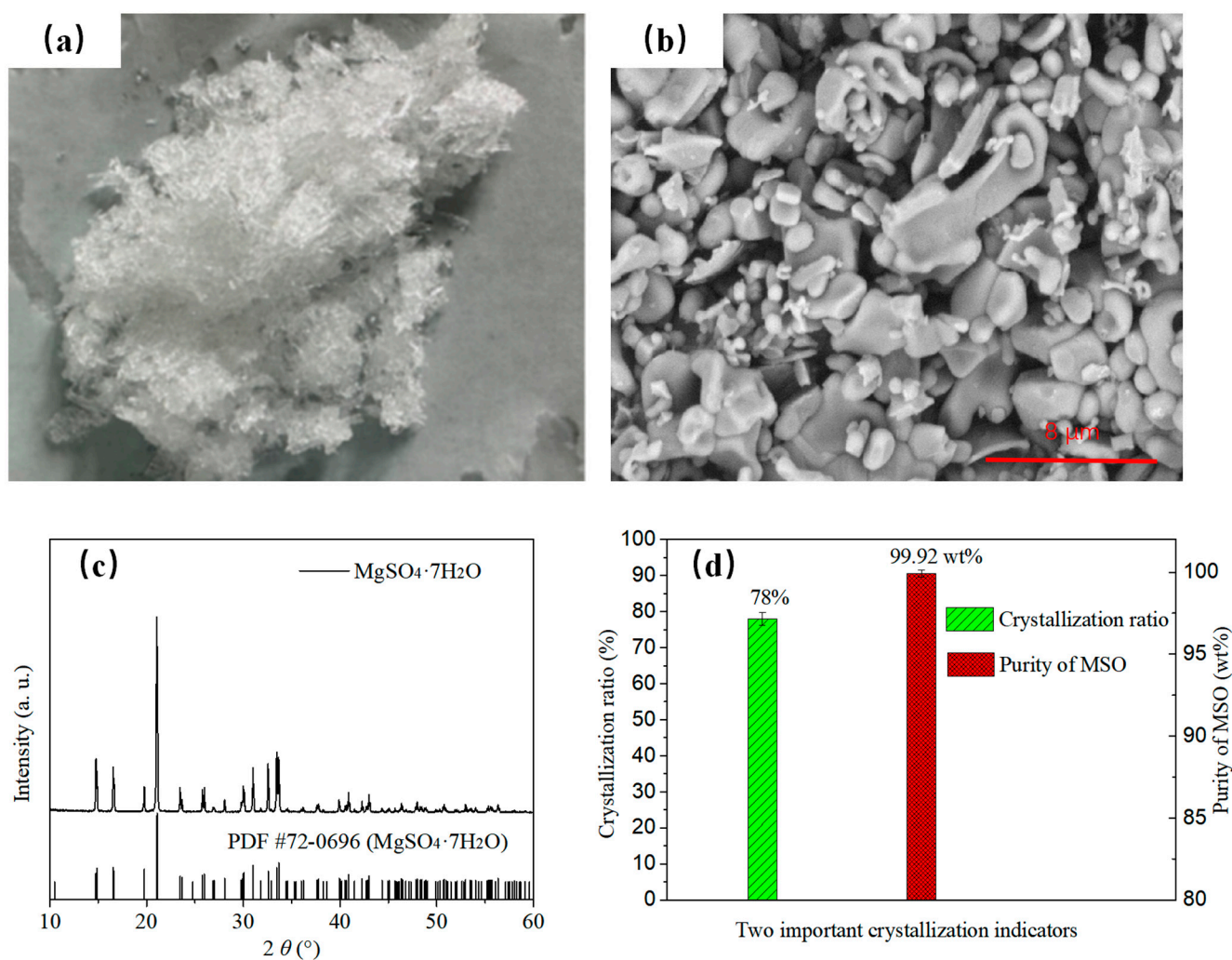


Figure 9. (a) Photograph, (b) SEM image, (c) XRD pattern, and (d) crystallization ratio and purity of the final MSO products.

3.6. Analysis of Synthetic Process for Portland Cement Utilizing Leach Residue as Raw Material

3.6.1. Analysis of Formation Process of the Clinker

Figure 10 shows the XRD pattern of the cement clinkers sintered at various temperatures. The results indicate that the clinker specimens of C1300, C1350, C1400, and C1450 consist primarily of C_2S , C_3S , along with a small amount of C_3A , C_4AF , and CaO phases. Well-developed C_2S , C_3S phases were observed in the C1400 and C1450 clinkers sintered at 1400 °C and 1450 °C, respectively. Among all the tested specimens, the C_2S

diffraction peaks of the C1400 clinker exhibited the highest intensity, whereas C1450 clinker presented the greatest relative content of C_3S , as evidenced by the intense diffraction peak at $2\theta = 32.14^\circ$, which corresponds to the strongest reflection of pure C_3S (PDF#49-0442). In contrast, the peak at $2\theta = 32.12^\circ$ matches the principal diffraction of C_2S (PDF#33-0302).

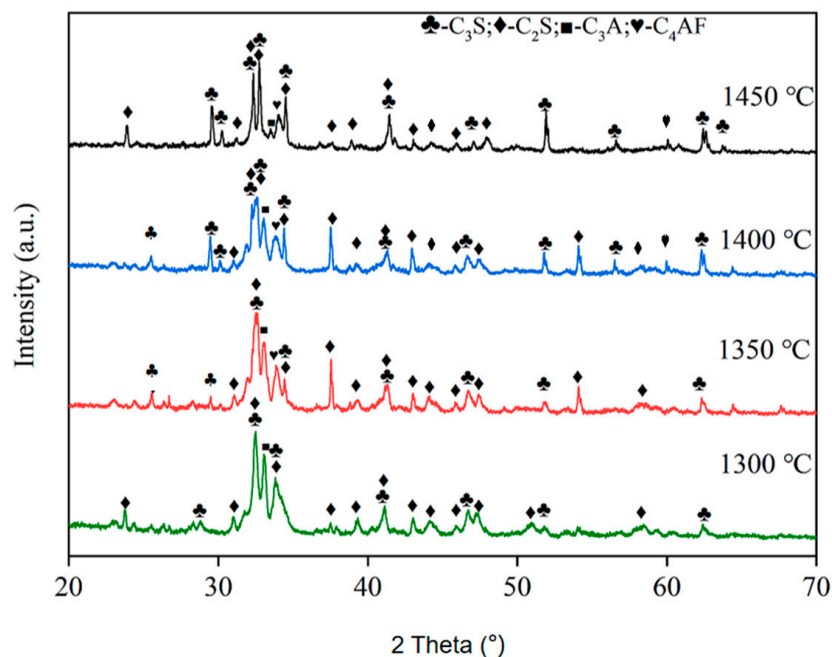


Figure 10. XRD pattern of the C1300, C1350, C1400 and C1450 clinkers.

Figure 11 displays SEM images of the C1350, C1400 and C1450 clinkers acquired at two magnification scales (300 μm and 20 μm), illustrating the effect of the calcination temperatures on the microstructure and phase development of the clinkers. As shown in Figure 11a–c, the sintered products exhibited a relatively loose structure with fine grains, and the interphase entanglement of different phases was more pronounced at a low calcination temperature of 1350 $^\circ\text{C}$. As the calcination temperature increased, the microstructure's structure became denser, grain size coarsened, and the interphase entanglement became less obvious. This evolution was attributed to the increased formation of the liquid phase at higher temperatures, which progressively filled the pores within the specimens, thereby enhancing structural densification and promoting more complete chemical reactions. High sintering temperatures facilitated the further transformation of residual unreacted or intermediate transitional phases into the target cementitious phases. Figure 11d–f show higher-magnification micrographs of the clinker specimens, revealing three distinct crystal-phase regions characterized by different contrast levels. For ease of description, these phases were categorized according to their relative brightness as black, gray, and white areas, combining the EDS scanning results of each spot in Table 6 and the XRD analysis results in Figure 10.

The black area at the first spot in Figure 11d shows a combined content of Ca and O elements of 90%, indicating that this domain corresponds predominantly to the free lime (CaO) phase. In addition, it contains a certain amount of Si and a small amount of Al elements, corresponding to C_2S and C_3A phases. The white area at the second spot reveals that the total content of Ca, Al, and O exceeded 85%, with C_3A as the dominant phase, along with trace amounts of C_2S , C_3S , or C_4AF . The gray area at the third spot exhibited a combined content of Ca, Si, and O elements exceeding 93%, indicating that this area is predominantly composed of C_2S and C_3S , with minor contributions from C_3A and C_4AF . Due to insufficient liquid-phase formation during calcination at 1350 $^\circ\text{C}$,

particle cohesion within this sample was weak. Furthermore, C_2S underwent the $\beta \rightarrow \gamma$ polymorphic transformation, resulting in volume expansion. As a result, there are a lot of pores in the sample. As shown in Figure 11b, when the calcination temperature reached 1400 °C, the black and white areas corresponding to the fourth and fifth spots decreased in size, while the gray area associated with the sixth spot expanded. The gray crystal phase evolved from the irregular polygons for the specimens sintered at 1350 °C into a relatively smooth, near-elliptical or rectangular morphology for the specimens sintered at 1400 °C, primarily composed of Ca, Si, and O, with a combined content exceeding 95%. This region corresponded to a mixed phase of C_3S and C_2S . The black area in Figure 11c was distinctly different from that in Figure 11a and closely resembled that of the gray area in Figure 11b. Moreover, its chemical composition was similar to that of the gray area in Figure 11b, indicating that the regions corresponding to the seventh and ninth spots in Figure 11c also consisted of a C_3S and C_2S mixed phase. Through further comparative analysis, we found that the molar ratio of Ca to Si was higher than that in Figure 11b, indicating an increased proportion of C_3S in the mixed phase formed at 1450 °C. In general, in the clinker sample sintered at 1350 °C, the phases of C_3S and C_2S did not fully develop, and the proportion of unreacted substances and intermediate products remained relatively high. As the sintering temperature further increased, C_3S and C_2S phases continued to grow and develop, becoming dominant crystalline phases in the cement clinker. However, these two phases are usually intimately mixed, making them difficult to distinguish morphologically. Further increasing the calcination temperature enhanced the proportion of C_3S with the ($C_3S + C_2S$) mixed phase. Meanwhile, no independent or fully developed calcium aluminate and ferroaluminate phases were predominantly distributed in the gaps between the mixed silicate phases.

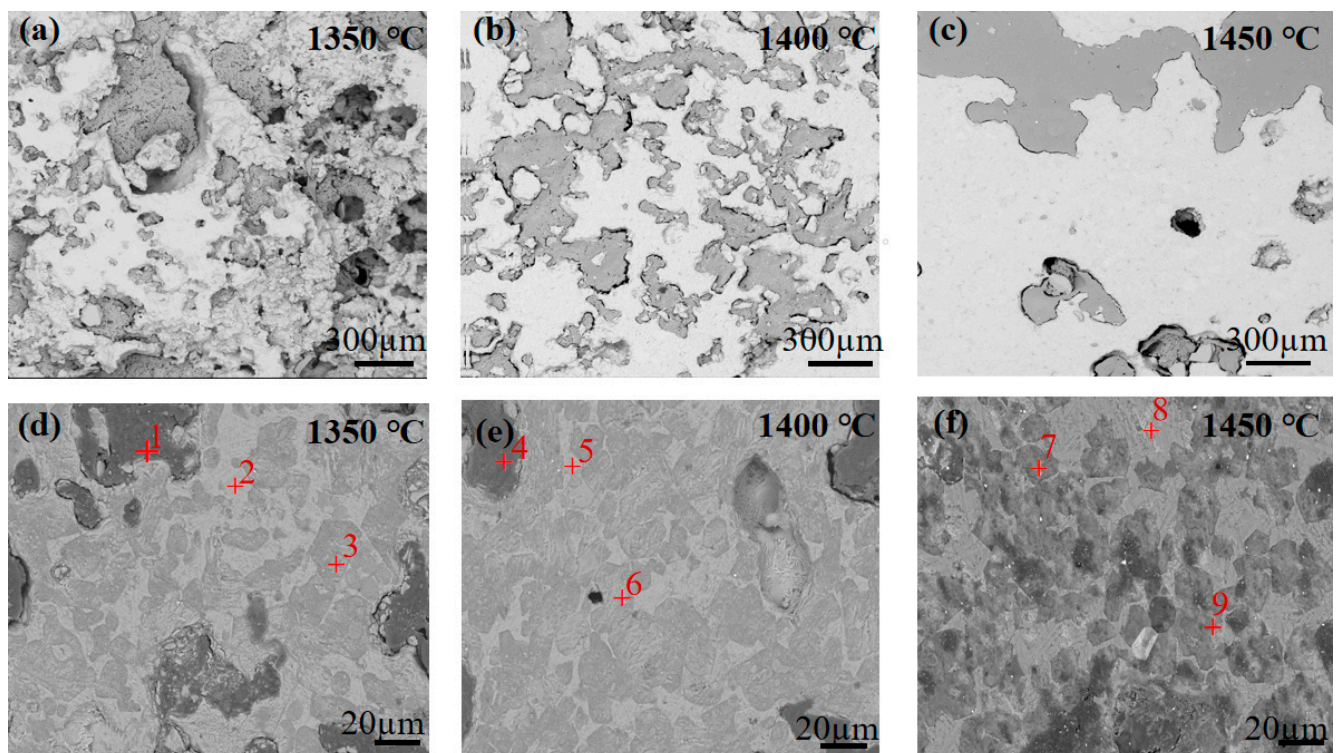


Figure 11. SEM images of the (a) C1350, (b) C1400, (c) C1450 clinkers at low-magnification scale, (d–f) at higher-magnification scale, respectively.

Table 6. EDS results based on the scanning measurement for the spots in Figure 11d–f (wt%).

<i>T</i> (°C)	Spot Number	O	Al	Si	Ca	Fe	Main Phase Components
1350	1	68.83	1.62	6.68	22.17	0.00	CaO and C ₂ S
	2	25.63	15.40	3.588	45.375	2.194	C ₃ A
	3	29.62	2.159	17.371	48.915	1.935	C ₂ S and C ₃ S
1400	4	33.30	1.965	14.645	45.732	4.354	C ₂ S, CaO
	5	25.67	11.35	5.355	45.258	12.359	C ₃ S, C ₄ AF
	6	32.18	2.303	14.550	48.615	2.353	C ₃ S, C ₂ S
1450	7	34.36	1.765	12.116	49.061	2.694	C ₃ S, C ₂ S
	8	26.50	16.16	2.580	43.232	11.521	C ₄ AF
	9	38.56	1.650	13.611	46.175	0.000	C ₃ S, C ₂ S

3.6.2. Property Analysis of the FNS Cements

Figure 12a presents the free CaO (f-CaO) content in the C1350, C1400 and C1450 cement clinkers, a key indicator of clinker burnability. With increasing sintering temperature, the f-CaO content in the clinker decreased linearly, demonstrating progressively enhanced burnability. For all FNS cement clinker specimens, the f-CaO content remains below 1.5%, satisfying the upper limit stipulated for Portland cement clinkers in Chinese national standards (GB/T 21372, 2008) [35]. At elevated sintering temperatures, greater quantities of low-melting-point silicate liquid phases formed, which accelerates the diffusion of heat and elements within the sintering system, thereby improving the kinetic conditions for phase formation. This improvement in kinetic conditions promoted more complete solid-state reactions, thereby reducing f-CaO content in the C1400 and C1450 clinkers.

Figure 12b shows the water demand of normal consistency for the C1400 and C1450 cement specimens, which are 29.56% and 29.47%, respectively. These values fall within the standard specified range of 27%–31% for Portland cement [38], confirming compliance with the international standard. The water demand of normal consistency is primarily governed by the quantity and relative proportion of the cement phases involved in the hydration reaction during cement setting and hardening processes. The slight difference between the C1400 and C1450 cements arose from their highly similar chemical composition and phase assemblages.

Figure 12c displays the setting times of the C1400 and C1450 cements, and the initial and final setting times were 97 min and 163 min for C1400 cements and 91 min and 165 min for C1450 cements, respectively. The setting times of the two types of Portland cement samples are very similar. Both satisfied the requirements of the Chinese national standard (GB/T 21372, 2008), which stipulates a minimum initial setting time of 45 min and a maximum final setting time of 390 min. The initial setting time of silicate cement is predominantly governed by the clinker's mineral composition, especially the content of alite (C₃S), as well as gypsum dosage, water-to-cement ratio, ambient temperature and humidity, and the presence of admixtures. Under identical conditions, a higher C₃S content accelerates the hydration reaction rate, thereby shortening the initial setting time. In contrast, the final setting time is chiefly influenced by the gypsum dosage and the content of C₃A and C₄AF in the clinker. Specifically, ettringite (calcium sulfoaluminate hydrate), formed by the reaction between C₃A and CaSO₄, acts as a delaying phase that delays the final set; meanwhile, the gelatinous hydration products of C₄AF formed a diffusion-limiting coating on the surfaces of clinker grain and further extending the setting period. Given their identical raw mix formulation and nearly identical phase assemblages, the C1400 and C1450 cements exhibit closely matched setting behavior.

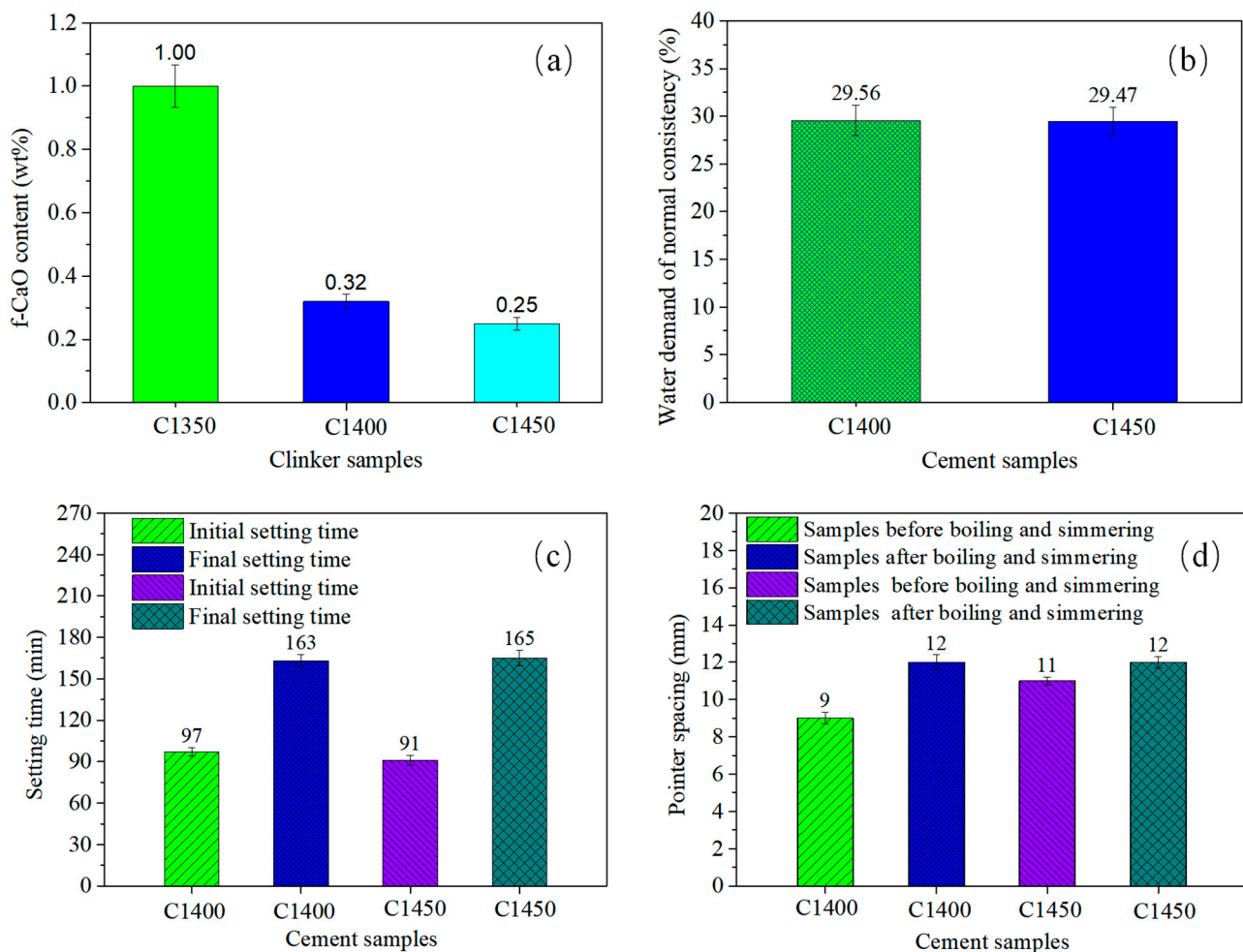


Figure 12. (a) f-CaO content of C1350, C1400 and C1450 clinkers, (b) water demand of normal consistency, (c) setting time, and (d) soundness of the C1350, C1400 and C1450 cements, with the clinker recipe of $KH = 0.82$, $SM = 2.04$, and $IM = 0.95$.

Figure 12d shows that the autoclave expansion values—quantified as average change in pointer distance before and after boiling—for the specimens of C1400 and C1450 cements, which are 3.0 mm and 1 mm, respectively. Both values are well below the 5 mm limit stipulated for Portland cement clinker in the international standard (ISO 9597, 2008 [38]), indicating that both Portland cements derived from sulfuric acid leach residue exhibited high stability under hydrothermal conditions. The presence of f-CaO and MgO in Portland cements can lead to hydration reactions upon exposure to water, forming corresponding hydroxides and releasing a large amount of heat, causing volume expansion and development of residual stress within the cement body, which adversely affects cement soundness. In severe cases, such expansion may cause deformation and cracking, compromising structural integrity and leading to poor dimensional stability. In the present C1400 and C1450 clinkers, the f-CaO content is low ($<1\%$, as shown in Figure 12a), and MgO primarily exists in the form of thermodynamically stable magnesium olivine (Mg_2SiO_4), which had a small influence on the soundness of the Portland cements, contributing to the good stability of the cements.

3.6.3. Analysis of the Hardened Products

Figure 13 shows the mechanical strength of the hardened products, which were prepared using the C1400 and C1450 cement clinkers, designated as C1400-mortar and C1450-mortar, respectively. The compressive and flexural strengths of the C1400-mortar af-

ter 3 days of standard curing were 35.63 MPa and 4.76 MPa, respectively, while after 7 days, they increased to 37.25 MPa and 7.98 MPa, and after 28 days, they reached 43.75 MPa and 10.32 MPa, respectively. The strength values of C1400-mortar met the minimum strength requirement for grade 42.5R Portland cement specified in the Chinese national standard (GB/T 17671-2021 [52]). In contrast, the C1450-mortar exhibited lower overall flexural and compressive strengths, with its strength performance satisfying the 32.5R grade classification under the same standard. A performance comparison between the hardened bodies of C1400 and C1450 mortars and the requirements specified in the standards is provided in Supplementary Table S2. Notably, the strength divergence between the two mortars is minimal during early hydration (3–7 days), reflecting comparable contributions from alite (C_3S)—the dominant phase responsible for rapid strength gain. However, after a 28-day curing time, the compressive and flexural strengths of the C1400-mortar exceeded those of C1450-mortar, attributable to the enhanced belite (C_2S) reactivity and greater cumulative hydration extent. As the principal contributor to later-age strength, C_2S hydrates more slowly but progressively, and its full contribution manifests only after extended curing, consistent with the observed strength gap widening over time.

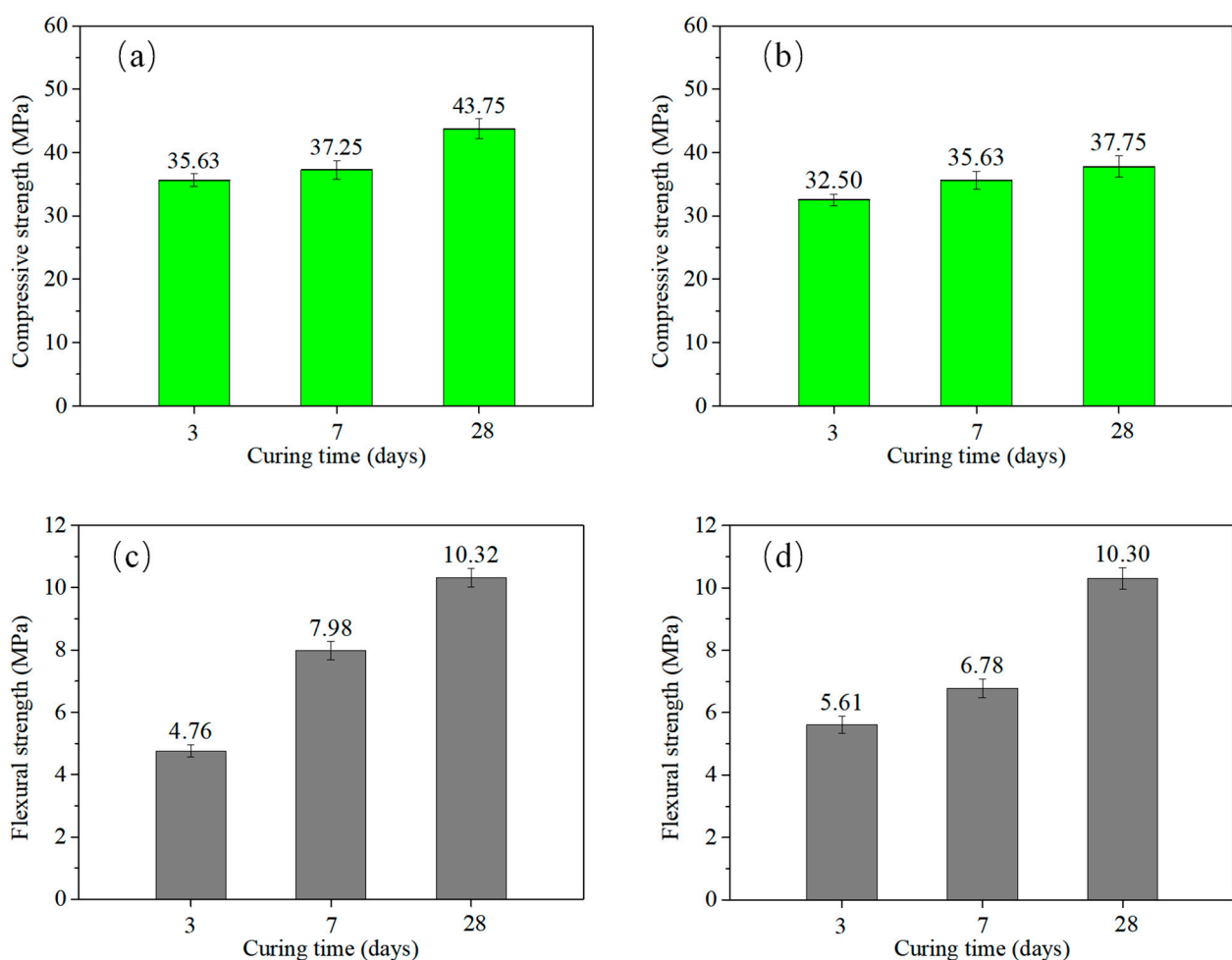


Figure 13. (a,b) Compressive strength, and (c,d) flexural strength of C1400-mortar and C1450-mortar hardened products, respectively.

4. Conclusions

- (1) Our leaching experiments demonstrate that the Mg leaching efficiency reached 90.75% when the milling duration of ferronickel slag was extended to 6 h in the absence of any co-solvent. XRD analysis revealed that the leaching slag consists of monopolistic-

phase forsterite (Mg_2SiO_4), with no detectable secondary crystalline phases. The leaching residue exhibited a marked decrease in MgO content and a substantial increase in SiO_2 content, making it more suitable as a silicon precursor in Portland cement production.

- (2) The neutralization–hydrolysis method has been validated as an effective purification strategy for removing impurities such as Fe, Al, Cr and Ni from crude magnesium sulfate solutions. During the process, MgO serves as an ideal neutralizer, effectively adjusting the pH of the crude magnesium sulfate solutions for the controlled hydrolysis and co-precipitation of impurity hydroxides. This pH-driven hydrolysis reduced the solubility of impurities without introducing extraneous impurity species into the system, thereby preserving solution purity and avoiding secondary contamination. Following secondary crystallization of the purified solution, the magnesium sulfate crystallization ratio reached 78.01%, and the purity of the resulting heptahydrate magnesium sulfate reached 99.92%, significantly enhancing its commercial applicability.
- (3) The leach residue obtained from FNS leaching is suitable as a silicon precursor in the preparation of cement clinker, and a balanced raw mix was formulated to achieve the target: $KH = 0.84$, $SM = 2.04$, $IM = 0.95$, incorporating approximately 23.95% leaching slag and 20.72% iron slag in the raw mix. The optimum sintering temperature for the cement clinkers is 1400 °C. The low f-CaO content (below 1%) indicates that the recipe containing leach residues exhibited good burnability. Results from mineralogical analysis and microscopic examination reveal that the C1400 and C1450 clinkers possessed well-developed cementitious phases. Furthermore, all performance parameters, including initial and final setting times, soundness, compressive and flexural strengths, of the C1400- and C1450-mortar met the requirements specified in the standards.
- (4) Mg loss occurred primarily in the leaching process, with the leach residue retaining approximately 10% of the initial Mg content. The remaining Mg loss was confined to the purification residue, retaining less than 3.5%. Mg^{2+} remaining in the post-crystallization mother liquor and the washing liquor of the leaching residue can be fully recycled within the magnesium sulfate production system, enabling an overall Mg recovery efficiency exceeding 85%. Upon incorporation of the leach residue into cement clinker production, the total utilization rate of the ferronickel slag approached 100%. A preliminary estimation indicates that, if this technology were implemented in industrial practice to utilize all the ferronickel slag produced in China annually, it would conserve at least 19.5 million tons of magnesite and 15.0 million tons of silica stone annually, while reducing CO_2 emissions by 10.3 million tons.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/su18052544/s1>, Table S1: The purity of magnesium sulfate products as stipulated in the national or industry standards of China; Table S2: Comparison table of main performance indicators of cement clinker and cement mortar of 32.5R and 42.5R products between standard requirements (ISO 29581-1, ISO 9597, ISO 679 and GB/T 21372, 2008 (in Chinese)) and the experimental results in this work.

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Abbreviations

The following abbreviations are used in this manuscript:

FNS	Ferronickel slag
MSO	$\text{MgSO}_4 \cdot n\text{H}_2\text{O}$
HP-MSO	High-purity magnesium sulfate
RKEF	Rotary kiln-electric furnace
IS	Iron slag
XRD	X-ray diffractometer
XRF	X-ray fluorescence
SEM	Scanning electron microscope
ICP-MS	Inductively coupled plasma mass spectrometry
LR-FNS	leached residue of ferronickel slag
<i>KH</i>	Limestone saturation coefficient
<i>SI</i>	Silica coefficient
<i>IM</i>	Alumina coefficient
C1400 clinker	The clinker sintered at 1400 °C
C1450 clinker	The clinker sintered at 1450 °C
C1400 cement	The cement prepared from C1400 clinker
C1450 cement	The cement prepared from C1450 clinker
C1400 mortar	The mortar prepared from C1400 cement
C1450 mortar	The mortar prepared from C1450 cement
C_2S	$2\text{CaO} \cdot \text{SiO}_2$
C_3S	$3\text{CaO} \cdot \text{SiO}_2$
C_3A	$3\text{CaO} \cdot \text{Al}_2\text{O}_3$
C_4AF	$4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{Fe}_2\text{O}_3$

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