

Review

Review on Comprehensive Utilization of Magnesium Slag and Development Prospect of Preparing Backfilling Materials

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Abstract: China is the largest producer of the metal magnesium, which is the third largest metal after steel and aluminum, and magnesium slag (MS) discharged from magnesium production cannot be treated effectively at present. Large amounts of MS occupy the land, making the land salinized and polluting the groundwater, which will threaten the sustainable development of the inland areas of China. To realize the large-scale utilization of MS, this paper reviews the research on the comprehensive utilization of MS and proposes using MS to prepare backfill materials. Firstly, the source and physical and chemical properties of MS are introduced, and the hazards caused by MS are also pointed out. Then, the several utilization methods of MS are summarized, such as cement admixture, chemically activated cementitious materials, clinker sintering, etc. Thirdly, the effect of MS on the properties of cementitious materials and concrete is summarized, including condensation time, workability, mechanical properties, durability, etc. Finally, based on the cemented backfill mining method, MS replaces Portland cement and blast furnace slag (BFS) to prepare cementitious materials. The mechanical properties and fluidity of backfilling slurry composed of MS, gypsum, BFS, and tailings can meet the requirements of backfilling mining.

Keywords: magnesium slag; resource utilization; physicochemical properties; backfilling mining; magnesium slag-based cementitious materials



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1. Introduction

Magnesium is the third most common metal after steel and aluminum. China is the largest producer of magnesium in the world, producing about 823,000 tons of metal magnesium in 2021, accounting for more than 85% of the global output. The Pidgeon process is the main method to produce magnesium in China [1]. However, MS is produced at a rate of 6~8 tons per ton of metallic magnesium. As a result, millions of tons of MS are produced in China every year, which cannot be effectively treated and is mainly stored and buried. Most of the magnesium smelting enterprises in China are positioned in the middle and upper reaches of the Yellow River. The arid climate in this inland area has resulted in a fragile ecological balance, and the threshold and resistance of the ecosystem are low. Large amounts of MS occupy land, make the land salinized, and pollute the groundwater. Those environmental issues have been threatening the sustainable development of the inland areas. In addition, the northwest district is the most centralized area for mineral resources in China, and large-scale mining action also destroys the local ecological balance.

This paper reviewed the research achievements on resource utilization of MS and pointed out the existing problems. Based on the cemented backfilling mining method, this paper outlines an approach to achieving large-scale utilization of MS. MS replaced the Portland cement and BFS to cement the solid waste, such as waste rock and tailings, and then the mixture stirred in the filling station on the surface was transported to the stope

through the pipeline. The backfilling body can maintain the stability of the stope and avoid the environmental issues caused by the mining process.

2. Sources and Physicochemical Properties of MS

2.1. Main Sources

The Pidgeon process is the main method to produce magnesium in China, as shown in Figure 1. Dolomite, as raw material, is calcined in the rotary furnace, the product is mixed with reducing agent silicon iron powder and catalyst fluorite, then ground and pressed into pellets, and the pellets are put into the reduction furnace. Under the condition of temperature 1200 °C and internal pressure of at least 13.3 Pa, magnesium appears in the form of steam, passes through the condenser, forms crystals, and is finally refined by solvent to produce magnesium [2]. The remaining material in the reduction furnace is the MS.

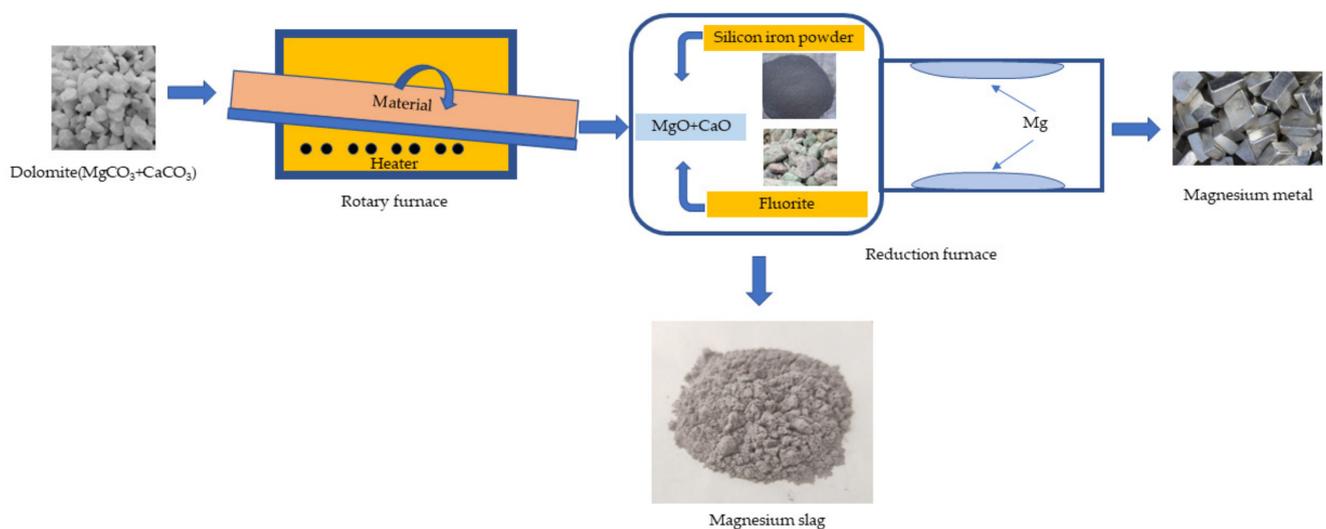
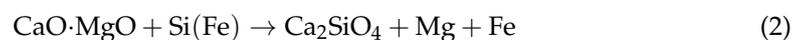
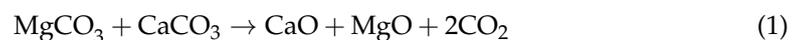


Figure 1. Flow chart of magnesium smelting by Pidgeon process.

MS is derived from the preparation process of magnesium metal, and the main chemical components of dolomite are MgO, SiO₂, Al₂O₃, Fe₂O₃, CaO, etc. Combined with the above analysis, the following chemical reaction occurred in the Pidgeon process:



The briquettes, which discharged from the retorts after the MgO reduction, contain the following two solid products: dicalcium silicate and Fe(s). During the cooling process of MS, Ca₂SiO₄, as the main product within the pellet, will undergo several phase changes. The change from β-phase to γ-phase, which occurs mainly between 400 and 500 °C. This change will occur in volume expansion, with an expansion rate of about 12% [3–7], and the solid pellet will disintegrate after expansion, producing MS particles [6,7].

2.2. Physical Property

MS is a gray substance, similar in color to cement, odorless, with a particle size between 0 and 10 mm, and slightly sticky to the touch. After experimental analysis, it was learned that [8], using the MS from a large magnesium smelting enterprise in Shanxi, the loose density was measured to be 1.15 g/cm³, and the average true density was 2.89 g/cm³. The magnesium residue is sieved, and the particle size distribution is shown in Figure 2 [9–12].

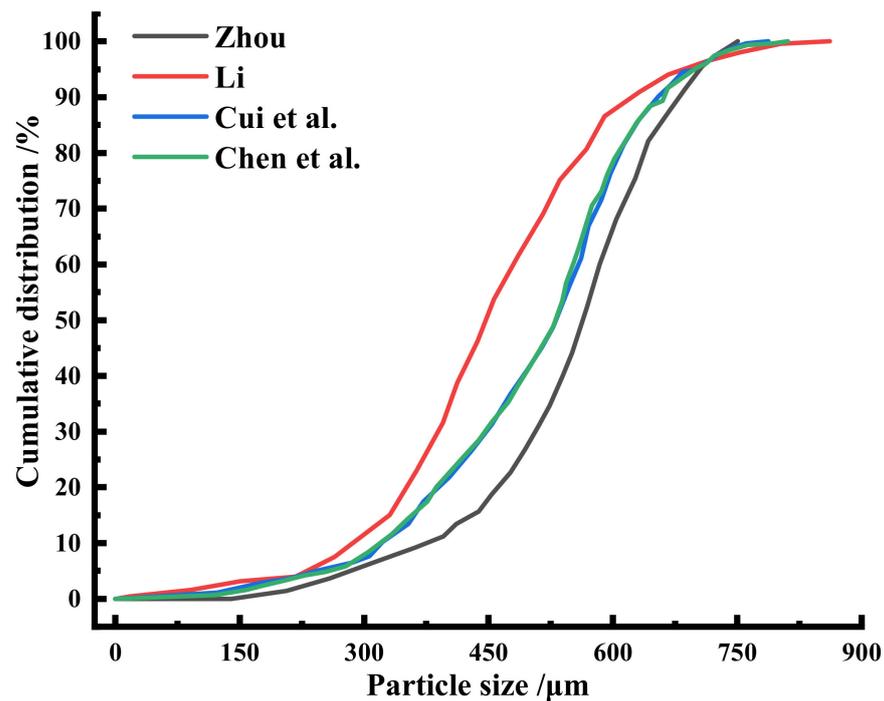


Figure 2. Particle size distribution of MS [9–12].

2.3. Chemical Composition

A total of 20 MS samples were collected from four plants producing metallic magnesium in Shanxi Province [13]. The main chemical components in MS are CaO, SiO₂, MgO, Al₂O₃, and Fe₂O₃. Djokic et al. [14] investigated the chemical compositions of twenty slag samples from the deposit of the Mg-Serbian Company. Han et al. measured the content ratio of CaO to SiO₂, i.e., the basicity, for the slag it is 1.58.

The quality evaluation parameters such as the alkalinity coefficient M_0 , the quality coefficient K , and the activity index M_a of MS were calculated according to the results of the chemical composition of MS, and the results were obtained in Table 1.

Table 1. Activity evaluation index of MS based on chemical component.

Source	M_0	K	M_a
Li, Y. et al. [13]	2.143	2.310	0.055
Djokic et al. [14]	1.667	2.997	0.499
Han, F.L. et al. [15]	1.773	1.795	0.023
Tengxiang Magnesium Products Co., Ltd., Hami City, Xinjiang Province, China	2.02	2.098	0.027

Among them, the alkalinity coefficients were all greater than 1.0, which means those MS all belong to alkaline slag powder. It was seen that those MS were classified as medium-quality slag powder as the quality coefficients were all greater than 1.6, and the MS has the potential to be prepared cementitious material. Although the activity indexes were all poor, the content of CaO, one of the main active ingredients, is high, 52.57%, 52.22%, and 42.35%, respectively, so it has potential activity and also can be used as an alkali-activator for cementitious material development.

The XRD pattern of the MS samples is shown in Figure 3. It is seen that γ -Ca₂SiO₄ (γ -C₂S) is the main phase that causes the decomposition of MS, in which f -MgO is the secondary phase, and β -Ca₂SiO₄ (β -C₂S) is a trace phase, which indicates that a small amount of β -C₂S can occur in the cooling slag particles.

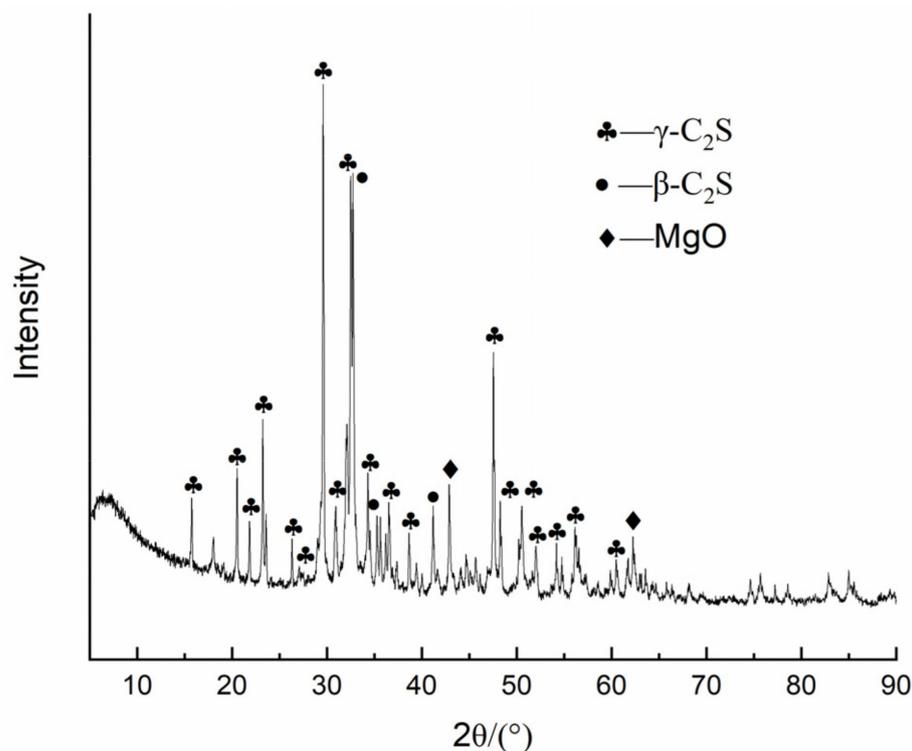


Figure 3. XRD pattern of MS.

The equilibrium phase distribution during the cooling process was obtained with the thermodynamic calculation using FactSage 6.2 [15]. At higher temperature conditions, $\text{Ca}_2\text{SiO}_4(\text{s}_2)$, such as α' - Ca_2SiO_4 polycrystalline, may only be 29% at 1000–1300 °C. $\text{MgOCa}_3\text{O}_3\text{SiO}_4(\text{s})$ as the main phase, such as merwinite formed by MgO, CaO, and SiO_2 , has a content of 47% at 650–1300 °C, but it will decompose in large quantities at around 500 °C, becoming the second phase in amount when the temperature is close to the room temperature phase, where the content is 17.5%. Therefore, $\text{Ca}_2\text{SiO}_4(\text{s})$, such as γ - Ca_2SiO_4 polycrystalline, will become the dominant phase when the temperature is close to room temperature, with a content of 56%. Any other slag with a similar content of γ - Ca_2SiO_4 will disintegrate into fine powder or dust.

2.4. Activity Excitation and Hydration Properties

MS has a chemical composition similar to cement, BFS, and other cementitious materials that contain γ - C_2S or β - C_2S and other mineral compositions with hydration properties; that is, MS has potential activity. Studies have found that [16–18], MS has certain gelling properties; the f -CaO and f -MgO in MS hydrate in the first. However, because the content of the early hydration mineral C_3S is lower, and the reaction rate of β - C_2S , the main gelling mineral in MS, is lower than that of C_3S , the weak hydration capacity of MS in the early-stage results in the low strength of cementitious materials.

2.5. Carbonization

MS contains a large amount of CaO and MgO, and calcium-containing components are mainly calcium silicate, lime, and silicate. Through CO_2 activation, Ca^{2+} and Mg^{2+} leach from Ca- and Mg-containing minerals can react with CO_3^{2-} and form calcium carbonate or magnesium carbonate, respectively. This dissolution-precipitation process is the carbonation of MS. After carbonation, part of the calcium carbonate is converted into calcite and sphalerite [19].

3. Hazards of MS

Magnesium production via the Pidgeon process is energy- and natural resource-intensive. There are several studies of environmental issues relating to magnesium production, including emissions of air pollutants and global warming impacts [20,21]. The global warming impact of magnesium production in China has been estimated near to 42 kg CO₂ eq/kg Mg ingot [21].

MS with fine particles is difficult to settle in the air and easy to cause dust pollution in the natural environment; long-term inhalation will lead to respiratory diseases. In addition, MS is the industrial solid waste from the smelting of metal magnesium; most of it will be directly dumped into the wasteland or landfill storage [22]. The harmful elements and heavy metal elements in the MS will penetrate the soil with surface precipitation, resulting in water pollution.

3.1. Harmful Components

During the smelting of magnesium metal, some harmful elements will remain in the MS and be enriched in the MS, such as arsenic (As), chromium (Cr), cadmium (Cd), mercury (Hg), copper (Cu), nickel (Ni), fluorooctane (F), and chlorine (Cl) [23].

The data on pollutants in MS in some regions of China were carried out by Wang et al. [23]. All the contents of F, Cd, Hg, Ni, Cu, and Cr in MS exceeded the values of China's soil environmental background and exceeded 18, 7, 6, 5, 4, and 2 times, respectively.

The hazardous substances contained in the MS are a threat to the safety of the water environment, and Wang et al. [23] determined the leachable concentrations of some heavy metals in the MS, including Hg, Cu, and Cr, whose extraction solutions exceeded the limit values (Class V) of the Groundwater Quality Standard (GB/T 14848-2017 2017), where the leachable concentration of F exceeded the limit values (Class IV–V). It shows that MS and MS-based fertilizers contain a variety of pollutant elements, which can leach into the ground by rainwater and surface water, thus posing a potential safety threat to soil quality and the water environment.

3.2. Dust Pollution

Studies have found that under stable weather conditions, MS dust particles will disperse around the reservoir for 1400–2000 m, and with climate change, their impact range will become wider [14].

MS is dust, accounting for about 60% of dust particles with a diameter of <150 μm. Because MS has a difficulty of settling out of the air, MS easily generates dust pollution in the natural environment and causes human respiratory diseases [23].

By adding an optimizing agent, one can effectively stabilize β-C₂S in MS and stop its transformation into γ-C₂S in the cooling process. Wu et al. [24] used several chemical stabilizers to treat the dusting MS at 1200 °C, including borates, phosphates, and rare earth oxides, and obtained volume-stable slag aggregates that were beneficial for environmental protection and recycling of the MS. After being taken out of the furnace, the sample of slag without stabilizers disintegrated immediately. The samples with stabilizer showed no dust at all, even after several months of removal. It was shown in X-ray diffraction patterns that the γ-C₂S is a major phase in the original MS sample, but its peaks are rather weak in the sample with chemical stabilizers, and the β-C₂S becomes a dominant phase. The research shows that the dusting of MS could be prevented effectively by additions of boric acid, calcium dihydrogen phosphate, and used cerium rouge [24].

4. Application of MS

As a common industrial solid waste, MS is widely used in cement admixtures, chemically activated cementitious materials, fertilizers, calcined porcelain, inorganic fibers, desulfurization, etc. in industrial production.

4.1. Cement Admixture

The poor hydration reactivity and safety risks of MS due to high levels of MgO inhibit its widespread use in the construction industry. However, MS has similar physical, chemical, and mineral properties as cement, so MS has potential uses as a mineral admixture in cement. Peng et al. prepared Portland cement by replacing clinker with two kinds of MS and investigated the properties of the cement, such as its water requirement for normal consistency, setting time, and strength. It was found that the addition of MS prolongs the setting time of cement. The cement meets the requirement of the P.C 42.5R standard when the additive content of MS ranges from 10% to 30%. While the additive content increases to 35%–40%, the cement can only reach the P.C 32.5R standard [25]. It was found that the activity of MS is slightly lower than that of BFS when they are mixed with clinker and gypsum to prepare composite Portland cement [26]. The flexural and compressive strengths of cement decrease as the content of MS increase when the content of MS and BFS fix at 50%. The properties of cement can reach the P.C 42.5 standard when the content of MS and BFS is 20% and 30%, respectively.

Studies have shown that MS has a greater impact on the physical and mechanical properties of the concrete, as the MS content increases the fluidity of concrete, and the fineness of MS also has a slight impact on fluidity and strength. Because MS is not conducive to the progress of the hydration reaction, the early strength will be affected but beneficial to the later strength. At the same time, the incorporation of MS optimizes the long-term performance of concrete, reduces dry shrinkage, improves sulfate, and increases freeze resistance [27,28]. The optimum content of MS as a mineral admixture in concrete can be up to 30% [29].

Soil stabilization is one of the methods of soil improvement in geotechnical engineering, of which cement and lime stabilization are the most commonly used methods in recent years and can reliably improve and increase soil strength parameters [30–34]. Through the analysis of the physicochemical characteristics and microstructure of MS, it is known that the addition of MS in the production process of cement can significantly reduce the production temperature, not only reduce the use of water, reduce energy consumption in the production process, shorten the condensation time, and, to a certain extent, make the cement have higher strength [28,29,35]. Through experimental tests, it was learned that as the content of MS and cement increased, the soil pH increased, there was a certain improvement in soil conditions, soil particles were granulated, and soil compressive strength increased [36].

The slaked magnesium slag (SMS) can be used as a blend that is suitable for the production of ordinary Portland cements. Because the MS is slaked, the SMS has no problem with soundness [28].

4.2. Chemically Activated Cementitious Material

Chemically activated cementitious materials in terms of either alkali-activated or acid-activated materials are of growing interest owing to their advantages over non-cement clinker, utilization of industrial wastes, low carbon emission, durable properties, or other special engineering performance [37,38].

MS containing high contents of CaO and MgO can be activated with CO₂ to prepare a calcium carbonate binder. The compressive strength of the MS paste reached up to 119.5 MPa after 14 d of CO₂ curing. This is mainly attributed to the formation of calcium carbonates, which leads to an enhanced product matrix with a dense microstructure in terms of reduced pore diameter and total porosity. Carbonation improves the volume stability of MS pastes [19].

It is known that alkali additions can activate pozzolanic materials to react and obtain strength. However, the strength of the mixture of MS and little clinker could not meet the engineering demand due to the lack of active ingredients. BFS was added to the cementitious system, and several activators were also used to improve the hydration

degree. The properties of MS cementitious material achieved the standards of composite cement of 32.5 MPa, and its hydration products are Aft, C-S-H, and Ca(OH)_2 [39].

4.3. Clinker Sintering

Most MS contains more than 50% (in mass) CaO, so it can be used as an alternative to lime-based materials for cement clinker production [40]. MS favorably improves clinker burnability with the introduction of MgO and CaF_2 . An appropriate amount of MS shortens the setting times of the cement and improves the compressive strength of the cement, allowing more C_3S to be produced in the clinker [40]. Heavy metals in clinkers can be immobilized or stabilized by hydrated matrices.

MS can be used to generate cement clinker, thus replacing part of the limestone, sand, and gravel at 11% [35]. The use of MS reduced the standard coal consumption and grinding time by 14% and 5%, respectively, and the results indicate that the use of MS can significantly improve the combustion properties and grindability and thus reduce energy consumption. The 28-d compressive strength of MS-based cement reached 58.9 MPa, and there was a significant increase in early compressive strength, but the setting time was relatively long, which may be related to the different crystalline states, and the physical properties of the cement using MS clinker met the requirements of the 52.5 strength class specified in GB 175-2007. The disposal of the MS via the production of Portland cement with the substitution of the raw material by MS is also environmentally safe.

The SMS used for the prevention of dust pollution was reactive. As a raw material, SMS can be used for calcining clinker of good quality [28].

4.4. Fertilizer

MS is rich in a large number of Ca, Mg, and other nutrients, which have a favorable impact on improving soil fertility and crop quality [41], so the further use of MS as fertilizer or improver in agriculture can not only effectively treat industrial solid waste but also achieve the comprehensive utilization of resources.

Related studies have shown that by mixing the MS with K_2CO_3 and heat treating it at 1300 °C for one hour, a novel slow-release potassium fertilizer was synthesized (SPF) [42], which could minimize fertilizer nutrient loss and Pidgeon magnesium slag (PMS) disposal problems. The active silicon content of SPF was 2.09 times as much as that of MS.

The silicon–potash fertilizer prepared from MS has great potential for application to Northern China's calcareous and nutrient-limited soil [43]. MS contains a large number of alkaline oxides (CaO and MgO), which would increase the soil pH and improve available K and Si absorption. The alkaline oxide in MS would react with K_2O and form semi-soluble aggregates during the preparation of Si-K fertilizer. The mobility of K^+ was decreased by incorporating it into the Si-O structure, and the solubility of Si was improved due to the depolymerization of Si-O tetrahedrons in the preparation of Si-K fertilizer.

MS, an alkaline byproduct during the refining of magnesium and magnesium alloys, contains some heavy metals (HMs) with a low content. Aiming to prevent soil HMs pollution in the application of MS-based fertilizer, the potential risk in the soil–plant system has been evaluated [44]. The results showed that the Cr, Cu, Pb, and Zn contents of two typical Chinese soil samples met the secondary standards of the Soil Environment Standard (GB 15618-2009), so the application of MS-based fertilizers to the two Chinese soils does not pose a risk of contamination to maize plants under current conditions. For more objective results, long-term field trials are needed to be conducted to verify.

4.5. Calcined Porcelain

With MS as a sintering aid, the sintering temperature of the proppant in the past was usually between 1300 °C and 1600 °C, which would produce huge energy consumption and bring high production costs [45–47]. By sintering MS with solid waste coal gangue at a sintering temperature below 1300 °C to prepare a low-density bauxite-based ceramic proppant, not only greatly reduced energy consumption but reduced production costs at the

same time. The studies showed that [48,49], at high temperatures in the sintering process, the MS reacts with the oxides in the raw material to form a liquid phase. Combining with the XRD patterns and SEM images, the main component of the proppant is granular corundum (Al_2O_3) and rod-shaped mullite ($\text{Al}_6\text{Si}_2\text{O}_{13}$). When the temperature increases, the intensity of the mullite diffraction peak increases significantly, and the corundum diffraction peak decreases significantly, which, due to the fact that the formation of the glass phase at high temperature provides favorable conditions for the good growth of the mullite's shaped nucleus and crystal nuclei, the proppant prepared by MS and gangue can form a fully glazed surface and a more compact structure at lower sintering temperatures.

4.6. Inorganic Fiber

At present, the market demand for thermal insulation materials is huge, and in order to effectively recycle a large amount of industrial solid waste, the preparation of slag mineral wool products has become an effective choice. However, as a short fiber, mineral wool cannot perform all the functions of fibrous material such as continuous basalt fibers. It is certainly the best choice that the continuous fibers are produced entirely from industrial solid wastes, both from the perspective of cost and resource utilization. Two inorganic continuous fibers were successfully produced by fly ash and MS with or without feldspar added, through melting at high temperature, following spinning into continuous fibers [50]. Their compressive strengths show a trend of first rising and then falling, with better thermal stability, and arrive up to 903 MPa and 539 MPa at room temperature, respectively. Two fibers also have excellent thermal resistance and stability.

It is known that any inorganic solid waste can be melted at high enough temperatures, but the preparation of the slag wool fibers needs to consider the cost and raw material source factors. One advantage of slag continuous fibers is the ability to artificial match various local slags and manage solid waste resources. It should be noted that the preparation of slag fiber must be considered to avoid secondary pollution.

4.7. Desulfurization

In MS, Ca exists not only in the form of CaO, but also in the mineral form of $\gamma\text{-C}_2\text{S}$ or $\beta\text{-C}_2\text{S}$, and it also contains a small amount of MgO. All this means MS may be used in the desulfurization process of coal-fired flue gas. However, the conversion rate of calcium used to evaluate the desulfurization performance of MS is very low when it is directly used for desulfurization.

The steam activation and quenching treatment methods are used to modify MS desulfurization activity, but the current performance is far from enough to meet the needs of industrial applications [51–53]. Compared with the naturally cooled MS, the particles of quenched samples are cracked and pulverized. The relatively rough surface made hydration easier by promoting reactant diffusion and dissolution. The degree of hydration increased with the increase in quenching temperature and hydration temperature. The calcium conversion rate of MS treated with continuous hydration is 30.3%, and the ones treated with non-continuous hydration are 13.3%.

(1) Through four modification methods of MS hydration, MS/fly ash hydration, MS quenching hydration, and MS/additive hydration to improve MSs desulfurization ability, their desulfurization performance is characterized by the calcium conversion rate. At the optimal parameters, the performance of the sample composed of quenching MS and fly ash hydration was the best. On the other hand, the additives such as $\text{H}_2\text{C}_2\text{O}_4$ and NaOH also enhance the MSs desulfurization performance, and the calcium conversion achieved was 73.7% and 55.54%, respectively.

(2) Based on the modified method of quenching hydration, additives such as NaCl, CaCl_2 , Na_2SO_4 , CaSO_4 , Na_2CO_3 , K_2CO_3 , acetic acid, and adipic acid were researched for improving quenched MS hydrate desulfurization performance. The 2% Na_2SO_4 -modified MS obtained the best result, and the calcium conversion rate reached 40.16%.

To sum up, combining various modification ways and several additives, the desulfurization performance of MS still cannot meet the requirements of industrial applications, and more relevant research is needed.

5. Effect of MS on the Properties of Cementitious Materials and Concrete

5.1. Condensation Time and Workability

The slump and slump flow of concrete increased as the content of MS increased from 0 to 40%. The improvement in workability was probably due to the filling effect and the relatively lower reactivity of MS. The MS filled in the voids between cement particles to displace the water and increase the amount of free water in the mixture. Besides, a lower content of water would be consumed by the cementitious materials incorporating MS during the early stages of hydration. All this would result in an increased amount of free water to improve the fluidity of the concrete containing MS [27].

As the additive amount of MS in the Portland cement increases, the amount of water needed for cement to retain its normal consistency increases, and both the initial and final setting times are prolonged. All of those are due to the high fineness of the powder and the strong water-absorbing properties of MS [54]. The addition of MS reduces the fluidity of cement without the water reducer, and the fluidity of cement increases with the increasing content of MS when the content of the water reducer is 2% of the cementitious material mass [55].

5.2. Mechanical Property

The compressive strength and the flexural strength of the concrete decreased as the amount of MS increased to 40% at all curing times. It can be known that the MS was not beneficial for the development of the early strength of concrete, but the MS gradually participated in the reaction to form calcium silicate hydration gel and improved the later-age strength [27].

The compressive strength of cement mixed with MS decreased rapidly with the increasing amount of MS. The negative influence of MS on early-period strength is greater than on later-period strength [54]. The 28-d and 60-d compressive strengths of cement paste added 10% MS reached 64.0 MPa and 76.4 MPa and increased by 10.54% and 14.37% compared with pure cement paste, respectively. It was indicated that MS granules dissolved and hydrated in the alkaline environment formed by cement hydration, pores between cement hydration products were filled along with the hydration of MS, which made the structure of cement paste more compact and obtained a higher compressive strength than pure cement paste [55].

5.3. Durability

The sulphate resistance properties of concrete can be improved by increasing MS content. After 150 cycles of sulphate attack, the sulphate-resistant coefficient of the reference sample (without MS) jumped down to 68%. The coefficient of concrete containing MS increased as the MS content increased [27]. This is due to the low reactivity of MS and the comparatively low Al_2O_3 content of MS. The number of hydration products that can react with SO_4^{2-} to form expanded products reduced after the MS replaced the cement [56]. In this case, the internal stress caused by expansion was reduced, and the sulphate resistance of the concrete was greatly improved.

5.4. Volume Stability

The incorporation of MS can effectively improve the resistance to drying shrinkage of concrete. The early drying shrinkage of the concrete increased with the increasing MS content in the early days. This is mainly ascribed that the MS reduced the amount of water involved in the hydration processing and increased the amount of free water in the mixture. The free water evaporation promoted the early shrinkage rate of concrete. At later ages, MS gradually participated in the hydration reaction, and the hydration products filled the

pores of the hardened concrete. As a result, the drying shrinkage of the concrete at later ages was significantly reduced by incorporating MS [27].

There are some percent of free CaO and MgO [6,7] that will hydrate in MS; the production of hydration can cause long-term volumetric instability for the slag [57,58]. The special and negative characteristics have made it rather difficult to use MS powder as building materials or aggregates for road construction [15]. The research shows that, by adding the borate agent and sintering at 1200 °C for several hours, the MS powder will be volume stable and can be used in building applications to replace valuable natural aggregates.

5.5. Hydration Heat and Degree

The total hydration heat of the blended cement paste decreased with increasing MS content. The appearance of the peak in the hydration rate curves was delayed by 4 h and 9 h for the blended cement paste containing 10% and 30% MS [27]. The reason was that the amount of C₃A and C₃S producing huge amounts of hydration heat at an early age was reduced with the replacement of MS.

Sun et al. found that the dissolution exothermic peak (the first exothermic peak) was reduced by the incorporation of MS, but the time of peak had not been affected. The addition of MS reduced the value of the hydration exothermic peak and delayed the time of peak, which positively correlated with the content of MS [55]. This study also showed that the hydration degree of cement added with MS decreased with the increasing of MS content; adding 10% MS did not inhibit the cement hydration, but the hydration rate and degree of cement with high content (50%) MS were seriously reduced.

6. Summary and Prospect

6.1. Large-Scale Utilization of MS

The recycling and utilization of MS has achieved great progress in the past years. However, the utilization amount of MS in practice is so limited that a large-scale utilization approach is urgent. The most potent way is to use MS to replace the Portland cement in backfilling mining.

Cemented backfilling mining method is the first option for mineral exploitation due to the recent stricter environmental protection policy in China. The expensive cost of backfill mining has put pressure on the state of enterprise operations, and the cost of cement is 70% of the cost of backfill materials. In addition, the tailings became finer along with the lower ore grade and the development of mineral processing technology. Portland cement has poor performance when ultra-fine tailings are used as aggregate, and its content needs to increase to achieve the same strength. So, it is urgent to exploit new cementitious materials adapted to the ultrafine tailings.

The properties of backfill materials had huge disparities with civil engineering materials, such as a higher water–cement rate, and lower strength request. Further, the shortage of cementitious materials prepared with MS cannot be accepted in civil engineering, but those can be allowed in the backfill mining and the required amounts of backfill materials are huge as more and more mines adopt the backfilling mining method. It is obvious that using MS to prepare backfill materials is the best way to realize large-scale utilization of MS.

Liu et al. modified magnesium slag with a chemical stabilizer and obtained a kind of modified magnesium slag (MMS) with high stability and good activity. Further, MMS and fly ash were mixed as cementitious materials with the aeolian sand as aggregate, and a new paste backfill material that has good mechanical properties and fluidity was prepared [59].

This paper utilized existing MS and industrial solid wastes to prepare cementitious materials for backfilling mining. The properties of cementitious materials composed of MS, gypsum, and BFS (MSGB) were researched. The MS used in this experiment was taken from Tengxiang Magnesium Products Co., Ltd., and it was ground to a certain fineness after drying to obtain higher activity. The particle size distribution of the MS was shown in Figure 4, the particle size characteristic parameters were listed in Table 2, and the chemical composition of the MS was listed in Table 1 in the form of three evaluation parameters.

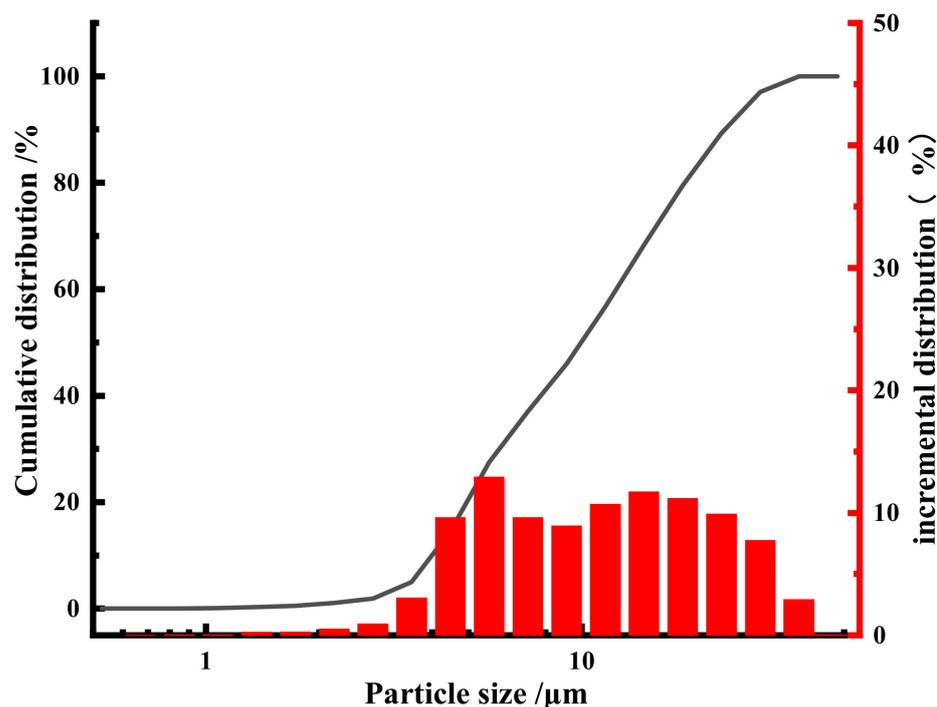


Figure 4. Particle size distribution of MS used in the experiment.

Table 2. Particle size characteristic parameters of MS.

$d_{10}/\mu\text{m}$	$d_{30}/\mu\text{m}$	$d_{60}/\mu\text{m}$	$d_{90}/\mu\text{m}$	Specific Surface Area m^2/kg
3.99	6.11	12.57	24.07	340

The level of MS was set at 25% and 30%; the level of gypsum was set at 4%, 6%, 8%, 10%, 12%, 14%, and 16%; the resting ingredient was BFS. Tailings taken from a mine in Xinjiang province were used as aggregate; the cement-sand ratio was 1:4, and the solid concentration was 72%. Portland cement was used as a reference. The strength of all samples was shown in Table 3.

Table 3. Strength of samples prepared with MSGB.

Number	Content of Cementitious Materials Ingredient/%			Strength/MPa	
	MS	GS	BFS	7 d	28 d
Y1	25	4	71	1.38	6.08
Y2	25	6	69	1.59	6.07
Y3	25	8	67	1.91	6.37
Y4	25	10	65	2.01	7.14
Y5	25	12	63	2.04	7.34
Y6	25	14	61	1.78	7.09
Y7	25	16	59	1.30	6.92
Z1	30	6	64	1.35	5.09
Z2	30	8	62	2.11	6.73
Z3	30	10	60	2.22	6.75
Z4	30	12	58	2.52	7.01
Z5	30	14	56	1.64	6.87
Z6	30	16	54	2.00	6.74
Z7	30	18	52	1.93	6.15
C1		P.O 42.5 100		1.97	5.29

It was shown in Table 3 that the 28-d strength of most samples prepared with MSGB was higher than P.O 42.5. The 7-d strength was equivalent to P.O 42.5, as the content of GS

is 8%, 10%, and 12%. Cement has a fast hydration rate and superior activity, and the sample can obtain better early strength. However, the abundant early hydration production will fill the pores of the sample and inhibit the diffusion of the hydration reaction, which is not conducive to the growth of later strength. In contrast, MSGB has poor activity and a low hydration rate, and the early strength of the sample is low. However, the hydration process of MSGB is long, and the late hydration rate can maintain a relevantly high level, which makes the sample have a higher late strength. Based on the above, it was feasible that MS blend can replace Portland and be used as cementitious materials in backfilling mining.

XRD is used to analyze the hydration products of MSGB (MS at 30% and gypsum at 12%) compared with cement, as shown in Figure 5. It can be seen that the hydration products with a diffraction peak in MSGB were ettringite, gypsum, unhydrated C_3S , C_2S , and MgO. The type of hydration products of MSGB is less than cement which hydration products contain $Ca(OH)_2$ and $CaCO_3$. The XRD patterns of MSGB and cement show a convex hull between 25 and 35° , which is the amorphous C-S-H gels. It is shown in Figure 5 that the diffraction peak of $Ca(OH)_2$ in cement and gypsum in MSGB decreases with the increasing curing age, which means that the $Ca(OH)_2$ and gypsum were constantly consumed in the hydration process and formed more hydration products. By that, the diffraction peak of ettringite in MSGB increased at 28 d curing age.

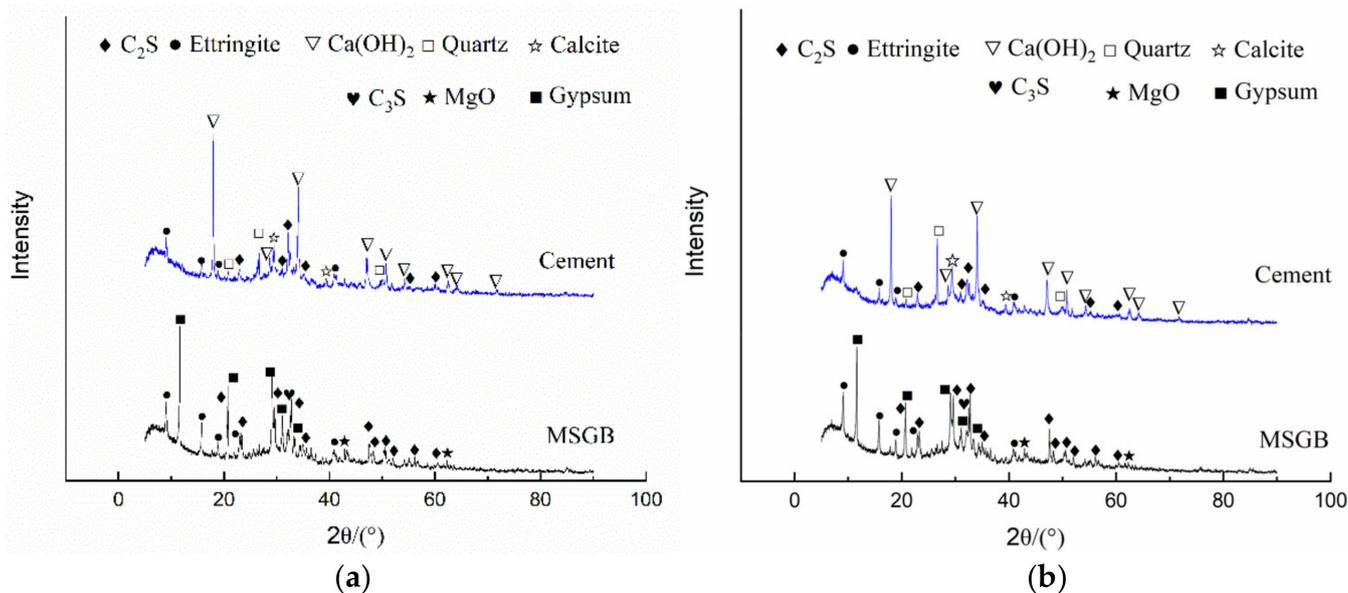


Figure 5. XRD patterns of MSGB and cement. (a) 7 d. (b) 28 d.

Figures 6 and 7 show the microstructure of the sample prepared with MSGB and cement under different magnifications at 7 d and 28 d curing ages. It can be seen in Figure 6a,c that the holes in MSGB are fewer than in cement, and that means MSGB has a more compact structure. Figure 6b shows that ettringite, which existed in the shape of a needle and rod interspersed in C-S-H gels, formed a dense network of structures. The structure of the sample prepared with MSGB was rather dense and hard to find holes in the surface at 28 d curing age, and the ones with cement still have many holes. It can be seen that the ettringite in the cement is still slim and short, and there are not enough C-S-H gels to connect the ettringite and form a compact structure. In contrast, it can be seen in Figure 7c that the surface of the sample with MSGB is completely covered with C-S-H gels, and ettringite is wrapped in that. The above can be used to explain at the microlevel why the strength of the sample with MGSB is higher than the ones with cement.

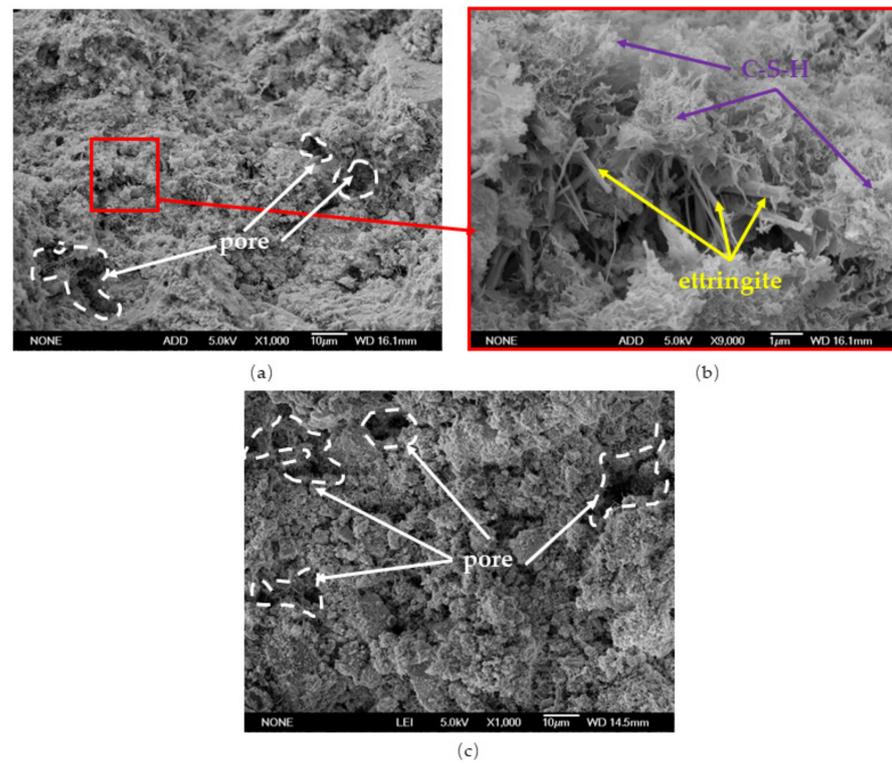


Figure 6. SEM images of MSGB and cement at 7 d curing age. (a) MSGB-X1000. (b) MSGB-X9000. (c) cement-X1000.

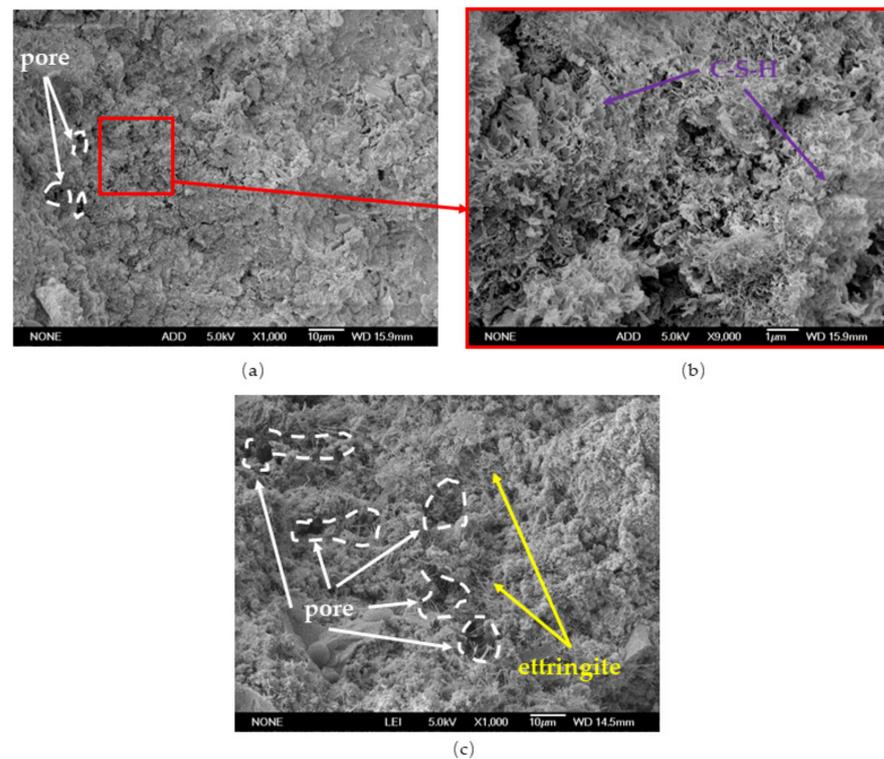


Figure 7. SEM images of MSGB and cement at 28 d curing age. (a) MSGB-X1000. (b) MSGB-X9000. (c) cement-X1000.

Strength and fluidity were the two critical factors to evaluate the performance of backfill materials. So, the rheological experiment of a slurry prepared with MSGB was

conducted with an R/S + SST rheometer at different conditions to evaluate its fluidity. The rheological experiment took the controlled shear rate test method (CSR), and the loading method was chosen as ramp loading, with the shear rate rising from 0 s^{-1} to 120 s^{-1} and then decreasing from 120 s^{-1} to 0 s^{-1} , the loading time of 240 s.

The rheological parameters were obtained through regressing test data based on the Bingham model and shown in Table 4. What can be clearly seen in this table is the decrease in slurry yield stress along with the decrease in the cement–sand ratio. Table 4 also reveals that there has been a sharp increase in the viscosity coefficient with the increasing of solid concentration.

Table 4. Rheological parameters of the slurry prepared with MSGB.

Solid Concentration/%	Cement-Sand Ratio	Yield Stress/Pa	Viscosity Coefficient/Pa·s	Correlation Coefficient
68	1:4	18.268	0.094	0.946
	1:6	15.070	0.107	0.959
	1:8	14.881	0.093	0.969
	1:10	13.401	0.091	0.946
70	1:4	32.430	0.177	0.966
	1:6	25.764	0.150	0.963
	1:8	25.681	0.149	0.964
	1:10	24.099	0.154	0.964
72	1:4	44.299	0.328	0.995
	1:6	43.032	0.327	0.996
	1:8	41.429	0.255	0.985
	1:10	41.060	0.276	0.989

In practice, pressure drop was widely used to estimate the pipeline transportation property of backfill slurry, which can be determined through a theoretical formula, L tube experiment, and pipe loop test. The pressure drop is related to the properties of the slurry (yield stress, viscosity, bulk density), the flow state, and engineering conditions [60]. The theoretical calculation is a more convenient and cost-effective method to predict the slurry pressure drop [61]. Before choosing the appropriate model, the Reynolds number Re shown in Equation (3) should be calculated to evaluate the flow state of the slurry.

$$Re = \frac{\rho v D}{\mu} \quad (3)$$

where μ is the plastic viscosity of the slurry, Pa·s; D is the pipe diameter, m; v is the flow velocity, m/s; ρ is the bulk density, kg/m^3 .

It was assumed in this paper that the pipe diameter is 100 mm, the flow velocity is 1.0 m/s, and all those parameters can be adjusted to match the backfilling technical conditions of specific mines in a practical application. The calculated Re ranged from 573 to 1960, as shown in Table 5, which means the slurry fell in the laminar flow regime ($Re < 2000$), and the Swamee–Aggarwal correlation as shown in Equation (4) was used to calculate the friction factor. Further, the friction head loss can be calculated by the Darcy–Weisbach equation and converted to a pressure drop i_m , and the calculation process has been introduced in reference [61]. The calculated pressure drop of the slurry is also shown in Table 5.

$$f = \frac{64}{Re} + \frac{10.67 + 0.1414 \left(\frac{He}{Re}\right)^{1.143}}{\left[1 + 0.0149 \left(\frac{He}{Re}\right)^{1.16}\right] Re} \left(\frac{He}{Re}\right) \quad (4)$$

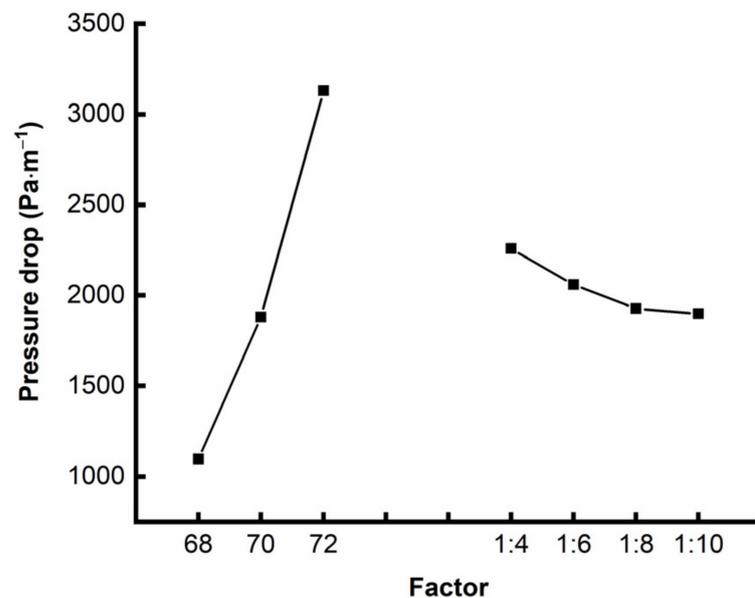
$$He = \frac{\tau \rho D^2}{\mu^2} \quad (5)$$

where f is the friction factor, τ is the yield stress; He is the Hedstrom number.

Table 5. R_e and pressure drop of the slurry at a pipe diameter of 100 mm, velocity of 1.0 m/s.

Solid Concentration/%	Cement–Sand Ratio	Yield Stress/Pa	Viscosity Coefficient/Pa·s	R_e	Pressure Drop /($\text{Pa}\cdot\text{m}^{-1}$)
68	1:4	18.268	0.094	1908.51	1227.62
	1:6	15.070	0.107	1671.03	1117.18
	1:8	14.881	0.093	1919.35	1058.91
	1:10	13.401	0.091	1960.44	979.02
70	1:4	32.430	0.177	1038.98	2216.07
	1:6	25.764	0.150	1220.00	1794.23
	1:8	25.681	0.149	1228.19	1786.61
	1:10	24.099	0.154	1187.66	1726.80
72	1:4	44.299	0.328	573.17	3330.55
	1:6	43.032	0.327	573.39	3264.15
	1:8	41.429	0.255	734.12	2934.19
	1:10	41.060	0.276	677.54	2989.84

Rang analysis was employed to compare the influence of solid concentration and cement-sand ratio on pressure drop, and the result is shown in Figure 8. It can be seen that the higher the solid concentration and cement-sand ratio, the higher the pressure drop, and the solid concentration has a greater impact on the pressure drop than the cement-sand ratio. The calculated results of pressure drop could be a reference for the design of the backfilling system, especially in the choice of pipeline transportation form (gravity flow or pumping).

**Figure 8.** Influence of solid concentration and cement-sand ratio on pressure drop.

6.2. Conclusions and Prospect

This paper introduced the sources, physicochemical properties, and environmental impact of MS, and especially summarized its application approach and effect on the properties of cement and concrete. The conclusion was obtained as follows:

(1) Based on the three quality evaluation parameters of MS, it is known that the MS is alkaline slag of medium- to high-quality and has poor activity. MS has the potential activity to prepare cementitious materials, and can also be used as an alkaline activator.

(2) The properties of cement can reach the P.C 42.5R standard and P.C 32.5R standard as the content of MS in cement ranges from 10%–30% to 30%–45%, respectively. However, clinker is still the necessary substance of composite Portland cement, whose content is 45% at least, and the application method that MS uses as cement admixture is still not

economically and environmentally friendly due to the clinker production process being energy intensive.

(3) Those application methods of MS, such as clinker sintering, fertilizer, calcined porcelain, inorganic fiber, and desulfurization, which all require high-temperature calcination and are also not economically and environmentally.

(4) The addition of MS will prolong the initial and final setting times of cement and increase the slump and slump flow of concrete due to the filling effect and the relatively lower reactivity of MS. The strength of cement and concrete decreased as the content of MS increased, and the negative influence of MS on early-period strength is greater than that on later-period strength. The hydration production of free CaO and MgO in MS can cause long-term volumetric instability, which limits the utilization of MS in building materials or aggregates for road construction.

(5) The special properties of backfilling materials, such as their higher water–cement rate and lower strength request, make it possible to realize the large-scale utilization of MS. The strength of the sample prepared with MSGB, composed of MS, gypsum, and BFS, was equivalent to or higher than the ones with cement. The pressure drops of a slurry prepared with MSGB are calculated through a theoretical formula, and the results can be used as a reference for the design of the backfilling system. The main hydration products of MSGB were ettringite, C-S-H, unhydrated C_3S , C_2S , and MgO.

In conclusion, MS blend can match the requirements of backfill materials both in strength and fluidity and has the capacity to replace Portland cement. The cost of MS blend, where the content of MS is 30% and that of GS ranges from 8% to 12% (the rest is BFS), is about 115–120 RMB/m³, including raw material cost, transport charges, and processing charges for grinding, and it is 68% cheaper than the cost of cement. Furthermore, the MS blend, as an alternative to cement, can vastly reduce the cost of backfill mining. Nevertheless, the following several issues should be researched in the subsequent work:

(1) The long-term mechanical property of the magnesium blend needs to be verified. Although the higher water–cement ratio of backfill can partly mitigate the negative effect of f -MgO in MS in the early curing time. The slow hydration rate and long hydration time of f -MgO would be harmful to the long-term strength of backfill, which has had a fixed internal structure. Furthermore, the gypsum as the activator and the hydration products ettringite all have expansibility. This effect will generate interstress on the pore, and affect the volume stability and strength of the backfill body.

(2) In addition to having activity, MS that contain f -CaO and f -MgO also can be used as alkali-activators to improve the hydration degree of pozzolanic materials. It is critical to find several low-cost activators that can be applied to actual industrial production to improve the activity of MS. Further, the interaction mechanism among the activator, MS, and pozzolanic materials needs to be researched.

(3) As previously mentioned, some harmful elements remain in MS in the process of smelting. The leaching property of cementitious materials prepared with MS must be researched, and guarantee the leaching rate of toxic components is lower than the requirement of relevant norms and standards.

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